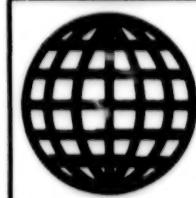


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JPRS Report

Science & Technology

***Central Eurasia:
Chemistry***

Science & Technology

CENTRAL EURASIA: Chemistry

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15 January 1992

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Moscow Oil and Gas Institute imeni I.M. Gubkin]

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[Abstract] The stabilization of surfactants is generally studied in specimens with a solid-phase concentration not exceeding several percentage points. Because suspensions with far higher concentrations are used in industry, the authors of the study reported herein investigated the characteristic features of the stabilization of diluted and concentrated suspensions over a broad range of concentrations. For their studies, they use precipitated barium sulfate with a specific surface (with respect to nitrogen) of $2.9 \text{ m}^2/\text{g}$ and chemically pure decane (manufactured by the firm BDCh). As a stabilizer they used the product ABS (manufactured by the Gorlov Chemical Combine) containing 80% (by mass) surfactant (alkylbenzene sulfonic acid). The methods of sedimentation analysis, refractometry, and laser diffractometry were used. Coagulation and peptization of the suspensions were studied by measuring the sedimentation volume at 25°. Refractometry was used to control both the initial concentration of surfactant in the liquid phase and its change upon adsorption. The coagulation studies were performed with initial concentrations of surfactant in the liquid phase of 0.04 to 0.30 g/l. The mass concentrations of the solid phase ranged from 4 to 26%. By establishing the fact of the formation of two types of adsorption films on the BaSO_4 grain surface, the authors were able to discover the molecular mechanisms of the stabilization of the study suspensions by surfactants. These two types of film, i.e., quasi-liquid and quasi-solid were deemed responsible for peptization of the solid phase. The formation of quasi-solid films was associated with markedly lower equilibrium concentrations of surfactant in the liquid phase than is the case when quasi-liquid films form. These results led the authors to conclude that controlled production of only quasi-solid films will make it possible to reduce the equilibrium concentrations of surfactant in the liquid phase. This result may be of practical value in a number of areas. When incorporated into the process of manufacturing drilling solutions, for example, it may result in a reduction of harmful active substances introduced into the environment. Figures 4; references 11: 7 Russian, 4 Western.

Contact Interactions Between Particles During the Dewatering of Colloidal Porous Capillary Peat Bodies

927M0012B Moscow KOLLOIDNYY ZHURNAL
in Russian Vol 53 No 3, May-Jun 91 (manuscript
received 25 Dec 90) pp 425-430

[Article by A.Ye. Afanasyev, Tver Polytechnic Institute]

UDC 532.685.539.4.01

[Abstract] During the process of structure formation and packing that occurs as colloidal porous capillary peat systems are dried, the strength (R_i) of the formed peat increases thanks to the effect of capillary forms, the number of coagulation contacts per unit cross section of specimens increases, and there is a reduction in the length of the intermolecular bonds between the elements of the structure coupled with the simultaneous organization of defects in it. Dried peat is thus subjected not only to natural physicomechanical defects but also to flaws that arise when it is dried and stored. In view of this fact, the author of the study reported herein developed a method for estimating the contact interactions between particles during the dewatering of colloidal porous capillary peat bodies that could be used to estimate the quality of dried peat. He analyzed the dependence of the strength of an individual contact (P_i) on the moisture content and temperature of the starting material and determined the maximum possible strength of a peat product after drying. He then proceeded to derive equations describing the process of structure formation through the change in P_i and an equation for use in estimating the molecular force constants of real peat systems (i.e., $A = P_i 12h_0^2/r_i$, where r is the particle radius and h_0 is the minimal distance between structural elements). The values obtained by the author based on the equations developed were in good agreement with data published elsewhere in the literature. As a result, he recommended his method of estimating the dependence of the strength of an individual contact as a function of moisture content and temperature (i.e., $P_i = f(W, T)$) and the maximum possible strength (R_{Ti}) as a way of predicting the quality of the finished product throughout various stages of the process of drying colloidal porous capillary peat bodies. Tables 2; references 18 (Russian).

The Adsorption of Mixtures of Nonionic Polymers and the Electrokinetic Potential of Aerosil Particles

927M0012C Moscow KOLLOIDNYY ZHURNAL
in Russian Vol 53 No 3, May-Jun 91 (manuscript
received 7 Aug 89) pp 431-435

[Article by A.A. Baran and N.S. Mitina, Institute of Colloidal Chemistry and Water Chemistry, UkrSSR Academy of Sciences, Kiev]

UDC 537.363+541.183.5:661.185.4

[Abstract] The adsorption of nonionic polymers by disperse bodies has been little studied to date. In view of this fact, the authors of the study reported herein examined the adsorption of selected nonionic polymers and their binary mixtures (1:1, 1:2, and 1:3) from water and electrolyte solutions by aerosil particles. The following chemicals were used in the study: Hi-Sil aerosil (specific

surface, 190 m²/g), polyvinyl alcohol with a mean viscosity molecular mass of 20,000 and 2% nonsaponified acetate groups, polyvinyl pyrrolidone with a molecular mass of 24,000, and methylcellulose with a molecular mass of 84,000. Adsorption was measured after 24 hours of contact between solution and adsorbent at room temperature (22 to 24°C). An equilibrium solution was separated from the sediment by centrifugation for 30 to 60 minutes at 20,000 rpm. The studies revealed that adsorption of the said polymers increases in the following sequence: polyvinyl alcohol < methylcellulose < polyvinyl pyrrolidone. Adsorption of the polymers and binary mixtures studied was found to result in a lowering of the electrophoretic mobility and ζ -potential of the particles. In a number of cases, adding binary polymer mixtures was found to result in an even greater lowering of electrophoretic mobility than did adding individual polymers. Data from adsorption experiments revealed that the sum adsorption of a binary polymer mixture is generally less than the adsorption of an individual component of the polymer mixture of the same concentration. This finding led the authors to conclude that the conformation changes occurring in the macromolecules in the surface layers are the result of the concurrent adsorption of polymers. Figures 4, table 1; references 9: 6 Russian, 3 Western.

The Theory of Aggregation in Colloids. The Nucleation Center Formation Stage

927M0012D Moscow KOLLOIDNYY ZHURNAL
in Russian Vol 53 No 3, May-Jun 91 (manuscript
received 23 Mar 89) pp 436-442

[Article by Yu.A. Buyevich and A.O. Ivanov, Ural University, Sverdlovsk]

UDC 541.18.04:536.423

[Abstract] Two basic approaches exist to the problem of describing the stratification of colloids and the formation of aggregates with a particle concentration different from that in the mother liquor. The first approach is based on an investigation of the kinetics of complexing or a loss of single particles by aggregates and is a natural extension of Smolukhovskiy's classic method. The second approach is based on an analysis of the properties of the equilibrium states of colloidal and micellar solutions by thermodynamics and statistical physics methods. According to the latter approach, aggregates are looked upon as a new phase resulting from a disturbance of the colloid's thermodynamic stability and has much in common with the stratification of molecular solutions. The main drawback of the said approach is its lack of a complete statistical-thermodynamic theory of colloids. In view of this fact, the authors of the study reported herein consider the problem of stratification of colloids by using a phenomenological thermodynamic model that was first described elsewhere. They examine

the equilibrium of spherical aggregates with a surrounding colloidal medium in the range of its metastability. They determine the minimal work required for spontaneous formation of nucleation centers and their critical size and those parameters characterizing the kinetics of the movement of nucleation centers into the transcritical range (i.e., their transformation into macroscopic aggregates). The mathematical model developed and analyzed includes 27 individual relationships or groups thereof. The authors emphasize that in order for their proposed model to be adequate from a physical standpoint, it is necessary to view the mother phase during the nucleation center formation process as an equilibrium phase whose properties are independent of the said process. They further emphasize that critical nucleation centers must, just as in the corresponding theory for molecular systems, be looked upon as macroscopic formations containing a rather large number of particles. This in turn requires that the critical nucleation center radius not be too small, which in turn makes it impossible to use the proposed model when analyzing states close to the boundary of the absolute thermodynamic instability and, especially, when analyzing states of actual instability. Figures 2; references 20: 11 Russian, 9 Western.

The Effect of Defects of the Active Layer of Reverse-Osmosis Membranes on Their Characteristics

927M0012E Moscow KOLLOIDNYY ZHURNAL
in Russian Vol 53 No 3, May-Jun 91 (manuscript
received 23 Aug 90) pp 443-448

[Article by V.D. Volgin, T.S. Nikitina, and S.M. Krivobok, Mir Scientific Production Association]

UDC 532.71:542.67

[Abstract] The performance characteristics of a reverse-osmosis membrane are determined primarily by the properties of its active surface layer. Defects in membranes' active surface layers as a result of the manufacturing process are inevitable. Because the role of such defects may not be obvious and thus may not be given consideration during research studies performed on membranes, the authors of the study reported herein set out to estimate the number of defects occurring in commercially produced Soviet reverse-osmosis membranes and to determine the role of these defects when membranes' characteristics are measured. They identified the defects present in four types of conventional Vladipor cellulose acetate membranes (MGA-70, MGA-80, MGA-95, MGA-100). A four-step identification procedure was used. In essence, developing solution was diffused through the membranes, which were kept in contact with photographic paper for 5 minutes each. An initial photographic print of the test membrane was obtained. The membrane's characteristics were then determined experimentally. A second photographic print was obtained, and the membrane's characteristics were

experimentally determined an additional time. The procedure used to produce the photographic print was found not to affect the membrane characteristics identified. The studies conducted showed that commercially produced reverse-osmosis membranes have numerous defects in the structure of their active layer and that these defects in turn have a great effect on the membranes' characteristics. Eliminating these defects, while difficult owing to the thinness of the active layer, will have a great effect on the membranes' performance. The studies further showed that when conducting theoretical research on membranes, researchers should use results of tests on membranes that do not contain a great number of defects. The authors recommended the method proposed herein to identify such membranes. Figures 5, table 1; references 4: 3 Russian, 1 Western.

Electrophoretic Mobility of Particles of Zirconium Dioxide Hydrosol

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in Russian Vol 53 No 3, May-Jun 91 (manuscript
received 21 Mar 90) pp 464-465

[Article by Dou Shen Yuan, V.V. Nazarov, and Yu.G. Frolov, Moscow Chemical Technology Institute imeni D.I. Mendeleyev]

UDC 546.831.4-31:537.363

[Abstract] Despite the numerous examples of the use of zirconium dioxide in alkoxide sol gel processes, data regarding the colloid chemical properties of zirconium dioxide dispersions are very limited and widely scattered. In an effort to remedy this problem, the authors of this concise report measured the electrophoretic mobility of particles of a zirconium dioxide sol produced by hydrolysis of zirconium isopropoxide in an aqueous solution of nitric acid followed by distillation of the isopropyl alcohol. The sol's electrophoretic mobility was measured by the microelectrophoresis method at room temperature with an electrical field intensity of 240 V/m and with platinum electrodes. The relative measurement error did not exceed 10%, and the concentration of zirconium dioxide in the sol amounted to 3% (by mass). In the zirconium dioxide concentration range studied (0.1 to 1.1 M), the articles of zirconium dioxide sol were found to carry a positive charge that increased as the acid concentration increased. The charge increase was attributed to adsorption of hydrogen ions on the surface of the sol's particles accompanied by the breaking of the Zr-O-Zr bond and the appearance of cationic forms of zirconium on the particles' surface that partially passed into the solution. A drop in the particles' electrophoretic activity was observed at HNO_3 concentrations above 0.9 M. The authors speculated that this drop was caused by the breaking up of the particles' surface layer. Figure 1; references 8: 3 Russian, 5 Western.

Toward a Theory of the Rheological Properties of Colloids

927M0012G Moscow *KOLLOIDNYY ZHURNAL*
in Russian Vol 53 No 3, May-Jun 91 (manuscript
received 11 Dec 89) pp 493-498

[Article by A.Yu. Zubarev, Ye.S. Kats, and A.N. Latkin, Ural University, Sverdlovsk]

UDC 541.18.04

[Abstract] The authors of the study reported herein have proposed a model of the orthokinetic coagulation of a concentrated suspension that predicts the nonmonotonic dependence of the effective viscosity on the deformation flow rate. Like the authors of a previous model of the relationship increases in a colloidal solution's flow rate and its viscosity, the model proposed herein considers the orthokinetic formation of aggregates containing a great number of colloidal particles that are assumed to be identical small spheres. A somewhat refined version of Smolukhovskiy's classic theory is used to study the earlier stage of the formation of doublets that are far removed from one another. To simplify their calculations, the authors assume that the aggregates are solid spheres that cannot be penetrated by the liquid. The authors conclude that their proposed model is valid if $uv \ll \rho_{\text{m}}$ (where ρ_{m} is the volume concentration of disperse particles in a dense packing), which in turn places constraints on the relationships between the energies U , T , and u (where U is the depth of the potential well of the pair interparticle interaction, T is the absolute temperature in energy units, and u is the energy acquired by each particle of the aggregate when it is deformed by an external flow). Figures 2; references 29: 18 Russian, 11 Western.

The Role of Surfactants in the Formation of the Charge of Particles in Stabilized Disperse Systems With a High Electrolyte Concentration

927M0012H Moscow *KOLLOIDNYY ZHURNAL*
in Russian Vol 53 No 3, May-Jun 91 (manuscript
received 4 Dec 90) pp 559-563

[Article by A.P. Tikhonov and V.Yu. Okorenkov Moscow Chemical Technology Institute imeni D.I. Mendeleyev]

UDC 535.36:532.773:541.183

[Abstract] Disperse systems with a high electrolyte concentration are widely used in various sectors of technology, including the production of composite electrochemical and electrophoretic coatings with specified properties. For this reason, the authors of the study reported herein examined the mechanism of the occurrence of a charge in similar systems. Their primary objective in so doing was to predict the systems' stability and, ultimately, the makeup and properties of the composites produced from them. The studies of the mechanism of surface charge formation were

performed by using an aqueous system with a high concentration of electrolyte containing diamond particles 0.5 to 1.0 μm in size (specific surface, $13.3 \text{ m}^2/\text{g}$). The system was stabilized by cationic surfactants with the following general formula: $[\text{C}_n\text{F}_{2n+1}\text{CH}_2\text{CH}_2\text{N}(\text{CH}_3)(\text{CH}_2\text{CH}_2\text{OH})_2]\text{Cl}^-$, where $n = 10$ to 12. The electrophoretic properties of the diamond particles were measured by the method of microelectrophoresis in a thermostatted plane-parallel cell. Copper reversible electrodes that were isolated from the working solution by plugs made of inert material were used.

An extremal dependence of the ζ -potential on surfactant concentration was observed. This dependence is determined by two factors. The first is the increase in the particles' charge on account of adsorption of the surfactant and the displacement of the glide plane to the boundary of the dense layer. The second factor is the reduction in the degree of dissociation of surfactant in the layer as it is formed as a result of the drop in the dielectric constant of the electric double layer. Figures 3, table 1; references 12: 11 Russian, 1 Western.

Determination of Specific Surface of γ - Fe_2O_3 from Adsorption Isotherms of Simple Gases

927M0021C Moscow ZHURNAL FIZICHESKOY
Khimii in Russian Vol 65, No 5, May 91 (manuscript
received 21 Apr 90) pp 1282-1287

[Article by B. V. Kuznetsov, Yu. S. Nikitin, T. A. Rakhmanova, and Ye. A. Simanovich, Moscow State University imeni M. V. Lomonosov]

UDC 541.183

[Abstract] Specific surface is one of the most important parameters in the production and study of acicular

γ -ferric oxide powders for magnetic recording. Frequently, the BET formula is used in conjunction with nitrogen or other gases as adsorbates. A problem arises in selecting a suitable size area for the adsorbate molecule in the adsorption layer, since it depends on the natures of both the adsorbate and the adsorbent surface. In the present work a vacuum scale was used to measure the adsorption isotherms of Ar, Kr, O₂, and N₂ at the boiling point of liquid nitrogen on two samples of acicular γ -ferric oxide designated for magnetic recording. The isotherms were then used to determine monolayer sizes of these adsorbates and the energy constants in the BET formula. Area sizes are presented for the above gas molecules during monolayer adsorption which may be used to determine the specific surface of γ -ferric oxide powders. Figure 1; references 13: 7 Russian, 6 Western.

Carbon Monoxide Reaction Mechanism with Co-Zeolite Catalysts

927M0021A Moscow ZHURNAL FIZICHESKOY KHIMII in Russian Vol 65, No 5, May 91 (manuscript received 19 Jan 90) pp 1200-1207

[Article by O. V. Ishchenko, N. V. Alekseyeva, K. P. Zhdanova, and F. K. Schmidt, Petrochemical and Coal Chemical Synthesis Institute at Irkutsk State University, Angarsk]

UDC543.422.4:541.183.5:546.735

[Abstract] Cobalt-containing high silicon zeolites are active catalysts in C_1 - C_{10} hydrocarbon synthesis from carbon monoxide and hydrogen. The present work is part of a continuing study of the active sites of Co-containing high silicon zeolite by adsorption of CO and hydrogen. Infra-red spectroscopy revealed that carbon monoxide is adsorbed on Co-NTsVM catalysts to form several types of surface compounds which are transferred to the surface complexes $Co^{+2}CO$, $Co^{+1}(CO)_2$, $Co^0(CO)_2$, and Co^0CO . The stability of these compounds varies with the degree to which the sample is reduced. Addition of manganese to Co-NTsVM enhances reduction of cobalt. The catalytic and adsorption properties of both catalysts, Co-NTsVM and Co-Mn-NtsVM, in hydrocarbon synthesis from carbon monoxide and hydrogen, are compared. Figures 3; references 12: 9 Russian, 3 Western.

Reactivation of a Modified Nickel-Molybdenum Catalyst for Glucose Hydrogenation

927M0024L Leningrad ZHURNAL PRIKLADNOY KHIMII in Russian Vol 64 No 3, Mar 91 (manuscript received 19 Sep 90) pp 684-686

[Article by A.G. Ivchenko, V.Kh. Khaysas, and M.F. Abdova, All-Union Scientific Research and Chemical Technological Institute of the Medical and Microbiology Industry]

UDC 541.127+542.941

[Abstract] In the past few years there has been a steady trend toward the use of continuous sugar reduction processes entailing the use of a modified nickel-molybdenum catalyst. For a number of reasons, the conventional method of regenerating coked catalysts, i.e., high-temperature annealing, is not suitable with catalysts for hydrogenation of monosaccharides. The authors of this concise report present the results of tests of a new method of reactivating spent modified nickel-molybdenum catalysts of glucose hydrogenation. The new method entails the use of solutions containing different concentrations of caustic soda. The caramelized catalyst was boiled at 90° in a 20% caustic soda solution amounting to twice the volume of catalyst for 3 hours. The catalyst was cleaned by a vibratory sieve and then rinsed. This was followed by the reactivation proper of the catalyst, which was accomplished by boiling it in a 10% alkali solution for 5 hours. Additional leaching of aluminum in an amount ranging from 10 to 12% occurred during the reactivation process. Microscopy studies revealed that upon reactivation by sodium hydroxide solutions, the catalyst began to form a grain structure characteristic of Raney's nickel. Tests of the reactivated catalyst demonstrated that its performance is comparable to that of freshly prepared catalyst (aluminum removal, 40%) under identical process conditions. The reactivated catalyst was equal to fresh catalyst with respect to both activity and stability. Table 1; references 4 (Russian).

Improvement of Process of Manganese Hydrophosphate [II] Production

927M0011A Kiev KHIMICHESKAYA
TEKHOLOGIYA in Russian No 5, Sept-Oct 1991
pp 18-21

[Article by V. A. Kopilevich; Ukrainian Agriculture Academy; Kiev]

UDC 546.71:546.185.325:541.484

[Abstract] Determination of conditions of production of individual magnesium hydrophosphate as the result of decomposition of manganese hydroxocarbonate by phosphoric acid involved the use of phosphoric acid and analytical grade commercial manganese hydroxocarbonate [II] containing 0.5-2.5 percent MnO_2 . Precipitation of Mn^2 ions with the use of $NaHCO_3$ or Na_2CO_3 at pH 6.9-8.2 produced manganese hydroxocarbonate with a low content of MnO_2 admixture. Calculations of the ratio of molar fractions of MnO , P_2O_5 , and H_2O in the gross formula and X-ray spectra identified the manganese phosphates. Use of freshly-precipitated manganese hydroxocarbonate produced a product yield of 99-99.5 percent of the theoretical level with a $MnHPO_4 \cdot xH_2O$ level of 99-100 percent in it. X-ray analysis of $MnHPO_4 \cdot xH_2O$ and $MnHPO_4 \cdot xH_2O$ suggested the identity of their structure. Changing the reaction temperature and conditions of production of manganese hydroxocarbonate may produce $MNHPO_4 \cdot xH_2O$ in addition to $MnHPO_4 \cdot xH_2O$. References 11: 10 Russian; 1 Western.

Optimization of Hydrogen Sorption Properties of Composites Based on Magnesium. 1. Effect of Degree of Interaction of Magnesium With Copper Hardener in Composite on Its Properties

927M0011B Kiev KHIMICHESKAYA
TEKHOLOGIYA in Russian No 5, Sept-Oct 1991
pp 22-26

[Article by M. M. Antonova, T. V. Khomko and V. A. Lavrenko; Institute of Problems of Study of Material; UkrSSR Academy of Sciences; Kiev]

UDC 669.017.3:669.721:621.787:669.3

[Abstract] One of a series of systematic studies of the stability of properties of composites at high temperatures included assessment of the hydrogen sorption properties of composites based on magnesium during its prolonged use under extreme conditions. The interaction of magnesium powder MPF-4 with electrolytically produced copper powder PMS2 in an 85:15 ratio was studied in the 250-500°C interval and the effect of products of the interaction on the hydrogen sorption properties of the composite were determined. Successful performance of the composite based on magnesium with a copper hardener required preliminary heat treatment of the composite at the earliest stages of the reaction of the copper with the magnesium. During prolonged use of the

composite, it was necessary to avoid conditions permitting passing of the reaction of the magnesium and copper up to complete combining of the copper in the intermetallic compound. The phase composition of the composite should be checked periodically by roentgenophase analysis. Figures 5; references 3 (Russian).

Multilayer Loading of Catalyst in Reaction Tubes of Natural Gas Vapor Conversion Ovens

927M0011C Kiev KHIMICHESKAYA
TEKHOLOGIYA in Russian No 5, Sept-Oct 1991
pp 27-31

[Article by L. A. Kadyrob, Yu. K. Bezuglyy and V. V. Veselov; Institute of Bioorganic Chemistry and Petrochemistry; UkrSSR Academy of Sciences; Kiev]

UDC 621.321:66.047.3

[Abstract] A study devoted to determination of some characteristics of the natural gas conversion process as a function of the nature of loading of the catalyst in reaction tubes and the possibility of repeat use of spent catalyst of conversion employed a mathematical model of a tube furnace for catalytic conversion of natural gas. Parameters of industrial operation of tube furnaces for vapor conversion of natural gas of ammonia synthesis aggregates with 450,000 tons per annum capacity served as starting data. Parameters affecting the duration of operation of reaction tubes were discussed and possibilities of increasing resistance of the catalyst to failure, reducing the cost and intensifying the natural gas conversion process were described. An increase of nickel surface in the catalyst layer reduced temperature indicators of the process, saved fuel and increased the efficiency of the oven. Increase of the mean nickel surface of the catalyst layer did not improve the technological parameters of the process studied. Figures 3; references 9: 7 Russian; 2 Western.

Analysis of Water Quality After Its Treatment in Immersion Combustion Apparatus

927M0011D Kiev KHIMICHESKAYA
TEKHOLOGIYA in Russian No 5, Sept-Oct 1991
pp 69-74

[Article by B. S. Soroka, A. S. Lukyanchikov, T. P. Stakhova and Ya. Ya. Derevyanko; Gas Institute; UkrSSR Academy of Sciences; Kiev]

UDC 662.951.2-213.32:66.048.54

[Abstract] Study of the effect on water quality of contact heat and mass exchange with products of combustion during bubbling during operation of a gas fueled immersion combustion device "Minaret", developed at the UkrSSR Academy of Sciences Gas Institute, involved determination of water quality according to 19 indicators in 28 water samples at different temperatures of heating and an excess of air feed to promote combustion.

Use of the contact method of heating water confirmed the possibility, advisability and safety for human health of use of the contact method of heating water. Use of the immersion heating device provided high uniformity of the temperature field along the liquid bath. Oxidizability, dry residue and alkalinity remained practically unchanged. Quality of water, heated by the immersion combustion device met sanitation and hygienic norms for swimming pools and household water supply systems. Figures 4; references 8 (Russian).

Production of Cellulose at Atmospheric Pressure

927M0011G Kiev KHIMICHESKAYA
TEKHOLOGIYA in Russian No 5, Sep-Oct 1991
pp 108-110

[Article by G. G. Kuryshko, A. A. Grigorenko, Ye. L. Matukhin and D. S. Nusinovich; Technological Institute of Light Industry; Kiev]

UDC 676.12.022.6

[Abstract] A study of the possibility of producing cellulose by cooking cotton lint irradiated by different doses of irradiation without pressure involved subjecting the cotton lint to a total radiation dose of 10-100 kGr and cooking at 100 degrees C for 2 hours. Preliminary irradiation of the lint produced cellulose with viscosity 100 times lower than that of cellulose produced from unirradiated lint, even at a 10 kGy dose. Reduction of viscosity proceeded slowly at 10-30 kGy but increase of the dose to 50 kGy or more caused marked reduction of viscosity of the cellulose produced. At a 10 kGy dose, the degree of polymerization of the samples studied equalled 756. Higher doses caused destruction of the lint and a very low degree of polymerization. Use of preliminary irradiation greatly reduced the cooking time and made it possible to perform the process at atmospheric pressure; thus simplifying apparatus set up and saving energy. References 3 (Russian).

Study of Possibility of Combining Hoisting and Lowering Operations in Drilling and Repair of Wells

927M0017A Moscow KHIMICHESKOYE I
NEFTYANOYE MASHINOSTROYENIYE in Russian
No 5, May 1991 pp 6-7

[Article by V. A. Ali-Zade, Candidate of Technical Sciences]

UDC 622.276.76:622.244.7

[Abstract] Possible combinations of the basic design of drill string hoist equipment components are described, and the influence of such factors as the type of pipe seizing equipment and whether individual sheaves of the block are mounted on the same or separate axes on the ability to combine individual components of drill string hoisting and lowering operations is analyzed. A diagram is presented of the trajectory of motion of the hoist block

with the drill string for various combinations and versions of hoisting and lowering operations with combined operations. The minimum time interval for a cycle of hoisting or lowering a single drill pipe stand depends on the design of each mechanism involved in the work. Analysis shows that the minimum time is achieved by combining the time involved in raising or lowering the unloaded hoist block with the time when a stand is shifted into or out of the axis of the drill string. Figures 3; References 9: 6 Russian, 3 Western.

Gas Liberation of New Materials and Coatings for Vacuum Cavities of Cryogenic-Vacuum Installations

927M0017C Moscow KHIMICHESKOYE I
NEFTYANOYE MASHINOSTROYENIYE in Russian
No 5, May 1991 pp 9-11

[Article by Ye. V. Chubarov, Candidate of Technical Sciences, Yu. V. Aleynik, L. A. Krasnoryadtseva and G. B. Sidorova]

UDC 533.5:621.52

[Abstract] The "Kriogenmash" scientific-production association has undertaken experimental studies of the gas liberation of corrosion-resistant type FP-545 and FL03-K coatings, used to protect the inner surfaces of low-vacuum chambers from corrosive media. The gas liberation of type TOV and TOV-M carbon-based black coatings containing polyvinyl butyral, as well as KO-818 coatings applied to aluminum cryopanels of protecting shields in vacuum chambers was also determined. Components of multilayer vacuum thermal insulation of surfaces cooled to cryogenic temperatures such as metallized polyethylene teraphthalate films, low-temperature paper, glass-reinforced paper and screen webs were also tested. The gas liberation of annealed aluminum foil was determined. Studies were performed at 1.33×10^{-3} Pa, 293 K with degassing continued over 150 hours using an oil-free evacuation system. The specific rate of gas liberation of materials as a function of time is illustrated. Gases liberated and the specific rate of gas liberation for various pumping times are presented in tabular form. Figures 2; References: 2 Russian.

Analysis of Effectiveness of Cryogenic Systems with Thermal Sorption Compressor

927M0017D Moscow KHIMICHESKOYE I
NEFTYANOYE MASHINOSTROYENIYE in Russian
No 5, May 1991 pp 12-14

[Article by A. P. Inkov, V. V. Redkin, Candidates of Technical Sciences, I. S. Komyanko, P. V. Nagornyy and G. I. Rogachev]

UDC 621.51.013

[Abstract] Thermal sorption compressors, the operation of which is based on the fact that metal hydrides can repeatedly and reversibly absorb and liberate large quantities of

hydrogen when cooled and heated, can increase the operating life of cryogenic microsystems to the 50,000 hour level due to the absence of any moving parts subjected to loads. However, this requires that all remaining components of the system, including the expander used to produce the cold in the low-temperature portion of the system, have equal reliability. A choke system with preliminary cooling is suggested for achievement of the temperatures needed for the use of high-temperature superconductors and various types of radiation sensors. Equations are derived for the exergetic efficiency of a system based on a thermal sorption compressor. Figures 4; References 5: 1 Russian, 4 Western.

Exhaust Pipe Models for Type GPA-Ts-6.3 Aviation Compressor Unit Drive

927M0017E Moscow *KHIMICHESKOYE I NEFTYANOYE MASHINOSTROYENIYE* in Russian
No 5, May 1991 pp 15-16

[Article by V. P. Parafeynik, A. M. Khoroshchenko, Candidates of Technical Sciences, S. D. Medvedev, V. Ye. Sukhinenco and M. N. Udom]

UDC 621.515-112-843.8

[Abstract] Results are presented from a study of a model of the exhaust pipe of an axial gas turbine of a converted type NK- 12ST aviation motor used in GPA-Ts-6.3 installations. Studies were performed on a static test stand to determine the total loss factor of the models in modes similar to the actual operating modes of the exhaust system. Models were studied in the range of $M = 0.42-0.52$ and $Re = (1-1.45) \cdot 10^6$. The use of an elongated model with reduced total losses can increase the efficiency of the motor by 0.33-0.35%, reducing the consumption of fuel by approximately 1.4%. The economic effect will be 4000 rubles per year per installation. Figures 3; References: 9 Russian.

Purification of Fluorine-Containing Gases in Fluidized Sorbent Bed

927M0017F Moscow *KHIMICHESKOYE I NEFTYANOYE MASHINOSTROYENIYE* in Russian
No 5, May 1991 pp 17-18

[Article by Z. V. Kuchma, T. I. Funatova, Z. G. Sash-
evskaya and A. M. Kossaya]

UDC 66.074.7

[Abstract] Results are presented from studies on the development and utilization of a dry method of trapping hydrogen fluoride in a fluidized sorbent bed. Primary attention was given to selection and evaluation of the sorption material, process conditions and arrangement of the purification system, as well as possible means of utilizing the spent sorbent. Materials tested included technical soda, phosphorite ore, chalk, limestone, calcium oxide, magnesium oxide, barium carbonate, aluminum oxide and fluorine-containing flux. The most

suitable materials are soda, alumina and barium carbonate. Spent sorbents can be used in the metallurgical industry and in the production of construction materials. Dry fluoride trapping systems can be successfully used in a number of production processes when the equipment for construction of such systems has been developed.

Optimization of Organosilicate Coating Composition for Protection of Metals from Ignition in Oxygen

927M0017G Moscow *KHIMICHESKOYE I NEFTYANOYE MASHINOSTROYENIYE* in Russian
No 5, May 1991 pp 21

[Article by N. I. Ulyanova, S. Ye. Narkunskiy, Candidate of Technical Sciences, Yu. I. Khudobin]

UDC 669.018-761:661.78

[Abstract] Coatings of organosilicate materials, the products of the chemical reaction of high-molecular-mass heteroorganic compounds with finely dispersed silicates such as asbestos, mica, etc. and metal oxides, are promising as fire-retardant coatings for steel and other materials used in oxygen equipment. This article studies the influence of individual components (silicates or oxides) in the materials on the combustion parameters, primarily ignition energy, and selects optimal compositions of organosilicate materials from the standpoint of fire safety. Compositions with 55-65% silicates by mass are recommended as the optimal compositions. The highest ignition energy was noted in a composition containing kaolinite.

Influence of Microarc Processing Conditions on Aluminum Alloy Friction Couple Dimensions

927M0017H Moscow *KHIMICHESKOYE I NEFTYANOYE MASHINOSTROYENIYE* in Russian
No 5, May 1991 pp 24-26

[Article by V. A. Fedorov, N. D. Velikoselskaya, Candidates of Technical Sciences]

UDC 621.794.61:669.715/621.891

[Abstract] Microarc oxidation is widely used to strengthen the surface of aluminum alloys by producing an oxide film, which is thicker than the original metal of which it is made. A study of the relationship of oxide thickness to original metal thickness indicates that the increase in thickness depends more on the microarc oxidation conditions (electrolyte composition) than on the type of alloy oxidized. An increase in current density during microarc oxidation intensifies the oxidation process, also increasing the thickness of the layer formed. Equations are derived allowing prediction of the thickness of the hardened layer formed as a function of processing time for various current densities. Figures 2; References 5: Russian.

Concentration Limits of Flame Propagation in Halogen-Hydrocarbon Vapor-Gas Mixtures

927M0021D Moscow *ZHURNAL FIZICHESKOY KHIMII* in Russian Vol 65, No 5, May 91 (manuscript received 24 May 90)

[Article by Yu. N. Shebeko, A. Ya. Korolchenko, O. Ya. Yeremenko, S. G. Tsarichenko, V. Yu. Navtsenya, and A. B. Ilin]

UDC614.841.12

[Abstract] Halogen-hydrocarbons, used widely in modern chemical industry, often include compounds capable of forming explosive vapor-gas mixtures such as methylene chloride, difluorochloroethane, tetrafluoroethylene, etc. However, the threshold limits of combustion of these substances, particularly the concentration threshold of flame propagation (KPR), have not been adequately investigated. In the present work KPR data were obtained on tetrafluoroethylene-difluorochloromethane mixtures, and a method is proposed for computing the upper KPR for combustible mixtures and non-flammable substances. This method must be further tested before being adopted for wide-scale use. Figure 1; references 14: 10 Russian, 4 Western.

Conflagration of Gaseous Mixtures of Hexafluoropropylene and Oxygen With Ultraviolet Light

927M0021I Moscow *ZHURNAL FIZICHESKOY KHIMII* in Russian Vol 65, No 5, May 91 (manuscript received 21 Jun 90) pp 1381-1383

[Article by I. R. Begishev and S. Yu. Smirnov]

UDC614.841.41+541.126

[Abstract] In evaluating the explosion hazard encountered in oxidation of organic compounds, and in deciding on appropriate safety measures, studies are made of mixtures of these compounds with oxygen to determine flash point, concentration limits of flame propagation, and other similar parameters. In the case of photochemical oxidation of such mixtures, these parameters may be affected by ultraviolet light. Short wave radiation is known to broaden the flash point limits of blasting powder. This, as well as hydrogen-oxygen mixtures, may be ignited with a UV-laser. The extent to which light is effective is determined largely by the capability of the system to absorb light and generate active particles. A mixture which absorbs light and also contains chlorine as oxidant, ignites readily under light at room temperature and atmospheric pressure. In the present work a study of the inflammation of a mixture hexafluoropropylene and oxygen at $0.4 - 3.0 \times 10^5$ Pa pressure demonstrates that the UV-flash point of this mixture decreases significantly in a vessel pre-treated with the reaction products. Addition of trace quantities of chlorine to the system, however, results in ignition at room temperature. Values for effective activation energies of the

process were determined and found to be different at high and low pressures. The surface characteristics of the reaction vessel also play a significant role. Figures 4; references 3: 2 Russian, 1 Western.

Quantitative Description of Rarefied Flames (H_2 or P_4CO_2)

927M0022A Moscow *IZVESTIYA AKADEMII NAUK SSSR: SERIYA KHIMICHESKAYA* in Russian No 6, Jun 91 (manuscript received 4 May 90) pp 1273-1296

[Article by Ye.N. Aleksandrov, Chemical Physics Institute imeni N.N. Semenov, USSR Academy of Sciences, Moscow]

UDC 541.124.7:541.126:546.11:546.18

[Abstract] This review summarizes the results of research on two branched-chain processes, i.e., the combustion of hydrogen and phosphorus close to the first combustion range. The discrepancy between the theory of these two combustion processes and the results of experiments examining the two processes is examined and attributed to heterogeneous factors that alter the combustion rate. The unusual (dual) first combustion range that appears under specified conditions upon the interaction of a rarefied flame and a surface with a complex composition is established and explained. Specifically, the following aspects of the combustion of hydrogen are examined: the conditions of the formation of an ultrarefined flame of an explosive mixture, the dependence of the nature of combustion and ultimate burnup on the state of the cold part of the reactor, the dependence of combustion induction periods on the surface state of the cold reactor space, self-inhibition of combustion under the effect of steam, and the heterogeneous negative interaction of chains and self-inhibition of reactions over quartz. The author proceeds to propose and test a quantitative mechanism of a rarefied flame of phosphorus and oxygen. The said mechanism gives consideration to the main (randomly caused) heterogeneous processes that either retard or accelerate the combustion process close to the first combustion range in the kinetic range of the breaking of the chains. These factors, many of which are caused by such random variables as the time taken to evacuate the reactor before the next test or the surface condition of the intake valves, are not considered in classical works on the topic. By taking these factors into account, the author is able to resolve some of the discrepancies between theoretical and experimental results. The second half of the review, which is devoted to the combustion of white phosphorus, covers the following specific topics: the reaction of oxygen atoms with a P_4 molecule, investigation of the reaction of atomic phosphorus with molecular oxygen, quantitative investigation of the reactions of PO with O_2 and PO with P_4 , quantitative description of a rarefied flame of phosphorus-oxygen mixtures, and the problem of coordinating schemes diagramming the phosphorus oxidation reaction with the dependence of the first combustion range on the concentration of starting reagents. Figures 14; references 44: 35 Russian, 9 Western.

Study of Conditions of Thermal Decontamination of Ethylacetate and Acetone in Boiler Furnaces

927M0011E Kiev KHIMICHESKAYA TEKHNOLOGIYA in Russian No 5, Sept-Oct 1991 pp 97-99

[Article by Yu. Pavlyuk, N. A. Gurevich, V. L. Aksenov and S. L. Zhukovskaya; Gas Institute; UkrSSR Academy of Sciences; Kiev]

UDC 502.55/203

[Abstract] A study of oxidation of ethylacetate and acetone vapors utilized a device consisting of a unit for preparing vapors of the organic substances, a mixing unit and an oxidation unit. The effect of the time of stay of the substance in the reaction zone, the concentration and temperature of the process were determined. The laboratory experiment showed the effective values of order of magnitude of the reaction and energy of activation: $n=0.4$; $E=188$ kJ/mole for ethylacetate and $n=2$; $E=196$ kJ/mole for acetone. The method of thermal decontamination in boiler furnaces proved to be effective for vapor-air mixtures containing concentrations of ethylacetate and acetone from 0.1-1.5 g per m³ and provided a high degree of purification. Figures 4; references 2 (Russian).

Installation For Utilization of Heat and Decontamination of Waste Gases

Kiev KHIMICHESKAYA TEKHNOLOGIYA in Russian No 5, Sept-Oct 1991 pp 99-101

[Article by N. V. Kostenko; Institute of Technical Thermal Physics; UkrSSR Academy of Sciences; Kiev]

UDC 662.99.004.8.002.73

[Abstract] A discussion of basic tendencies in development of methods of and devices for using the heat of escape gases included descriptions of devices using a high-temperature intermediate heat-transfer agent in a mixture of high-boiling and low-boiling components and a vortex tube with integrated peripheral zones which makes it possible to create a cold flow with a temperature meeting sanitary requirements and a hot flow with higher parameters than those found in an ordinary vortex tube which makes it possible to use it for heating without additional expenditures.

An Investigation of the Mechanism of Extracting Components of Acidic Sewage in the Process of Galvanocoagulation Cleaning

927M0024D Leningrad ZHURNAL PRIKLADNOY KHIMII in Russian Vol 64 No 3, Mar 91 (manuscript received 10 Apr 90) pp 551-555

[Article by L.P. Sokolova, Ye.S. Smurova, Ye.B. Kokorina, A.K. Samsonov, and Yu.M. Dedkov]

UDC 628.34

[Abstract] The method of galvanocoagulation cleaning, which was developed at the Kazmekhanobr [Kazakhstan Institute of Machining], is beginning to gain popularity as a way of neutralizing metal- and chromium-containing sewage. The method is based on using the effect of a galvanic iron-coke or iron-copper element placed in the solution being cleaned. Thanks to the difference in electrochemical potentials, the iron is polarized anodically and passes into the solution without the application of current from an external source. The coke or copper in the galvanic pair is polarized cathodically. The process is simple to implement and is carried out in drum-type flowthrough devices. Significant quantities of calcium and sulfate ions are also extracted from the sewage solution during the galvanocoagulation cleaning process. The authors of the study reported herein studied the mechanism by which components are extracted during the said process. They used iron-containing pellets and casting coke in a 4:1 ratio. Infrared spectroscopy, x-ray crystallographic, and x-ray phase analyses were performed to determine the phase composition of the starting pellets and three additional types of specimens: pellets treated for six cycles of sorption of nonferrous metal ions from a solution of acidic sewage, sediment from cleaning an iron pellet-coke galvanic pair with process water, and sediment formed during the cleaning of acidic sewage in an iron-coke galvanic pair mode. The studies performed demonstrated that the main mechanism of the extraction of ferrous metal and chromium ions from a sewage solution during the galvanocoagulation cleaning process in an iron-coke galvanic pair mode is that of the formation of nonferrous metal ferrites by means of the interaction of the nonferrous metal ions with oxide compounds of iron (particularly magnetite). Sulfate ions are extracted by interaction with hydroxide iron compounds with the formation of a complex compound containing Fe (III), SO_4^{2-} , and OH ions and water molecules. Another mechanism of sulfate ion extraction is that of its reduction to S^{2-} and separation in the form of slightly soluble iron sulfide (pyrrhotine). Intensifying sulfate ion extraction requires having a sufficient quantity of hydroxide iron compounds in the sediment. The most effective sulfate ion extraction is observed when the pH of the solution being cleaned is at least 2.5. Iron-containing pellets were found to be a more effective component for the iron-coke galvanic pair because they consist of magnetite and α -iron. Tables 3; references 7: 6 Russian, 1 Western.

The Use of Water-Soluble Alcohols To Extract Phenol From Aqueous Media

927M0024F Leningrad ZHURNAL PRIKLADNOY KHIMII in Russian Vol 64 No 3, Mar 91 (manuscript received 19 Apr 90) pp 573-577

[Article by Ya.I. Korenman, T.N. Yermolayeva, and T.A. Kuchmenko]

UDC 542.61:547.56.1

[Abstract] The development of new techniques to extract phenol from sewage and natural waters remains a critical chemical/ ecological problem. Oxygen-containing extractive reagents have proved to be the most effective phenol extractors. For several reasons, however, they have only recently begun to be used to extract organic compounds (including phenol) from aqueous media. In view of this fact, the authors of the study reported herein conducted a study to determine the optimal conditions for using propyl and isopropyl alcohols to extract phenol from aqueous-saline solutions. Ammonium sulfate and potassium carbonate were used as salting-out agents. The degree of phenol extraction [$R, \%$] was found to be significantly dependent on the pH of the aqueous phase. Highly acidic media ($pH < 2$) were found to best promote the water solubility of the alcohols used. The optimal degree of phenol extraction and precision of establishing the phenol distribution coefficient [D] were achieved when the pH of extraction systems containing a minimum amount of salting-out agent was kept between 2 and 5. Phenol extraction in systems with a maximal content of salting-out agent was found to be best with a pH of 1 or 2. D and R were found to be influenced not only by the pH of the solution but also by the nature of the acid added to the aqueous solution to create the specified pH. Sulfuric acid was recommended because it was found to increase the total concentration of sulfate ions in the solution and, thus, D and R . Ammonium sulfate and potassium carbonate were found to salt-out the alcohols in different manners. A lesser amount of potassium carbonate (as opposed to ammonium sulfate) was required for formation of an independent organic phase. It was discovered that by reducing the water solubility of the extractive reagent, the salting-out agent reduces the solubility of phenol in water and facilitates its transition into an organic phase. The degree of phenol extraction in systems with both propyl and isopropyl alcohols increases as the concentration of salt in the system increases and is highest in solutions saturated with salting-out agent. These findings led the authors to recommend extraction systems providing a high coefficient of phenol distribution and high degree of phenol extraction from aqueous media. Figures 2, tables 2; references 10 (Russian).

The Possibility of Increasing the Protective Power of a Lime Chemical Absorbent

927M0024G Leningrad ZHURNAL PRIKLADNOY KHMII in Russian Vol 64 No 3, Mar 91 (manuscript received 13 Feb 90) pp 578-582

[Article by G.K. Ivakhnyuk, T.G. Kozhina, V.V. Samonin, N.F. Fedorov, and M.O. Slesareva]

UDC 661.183

[Abstract] A great deal of research has been devoted to developing the pore structure and optimizing the moisture retention properties of protective lime chemical

absorbents. The anticipated improvements in the said absorbents' protective characteristics have yet to be achieved, however. In view of this fact, the authors of the study reported herein undertook their own research to improve the protective properties of lime chemical absorbents. After analyzing the existing literature on the topic, the authors hypothesized that lime chemical absorbents could be improved if subjected to chemical modification by water-soluble hydrophilic polymers. They used the following water-soluble polymers as chemical modifiers of the formulation of lime chemical absorbents: polyacrylamide, alkaline metal salts of polyacrylic acid with various degrees of neutralization of the carboxyl groups, and selected complexing agents. Specimens of lime chemical absorbents modified by each of the said modifiers were prepared according to the familiar method. The prepared specimens were subjected to comparative tests relative to a commercially manufactured lime chemical absorbent until a layer with a breakthrough concentration of CO_2 in the gas-and-air mixture equal to 0.1% (by volume) under the following dynamic test conditions: concentration of CO_2 in the gas-and-air mixture, 2.0% (by volume); flow rate of the gas-and-air mixture, $0.3 \text{ l}/(\text{min} \times \text{cm}^2)$; moisture content of the gas-and-air mixture, 87%; temperature of the gas-and-air mixture, 20°C ; and length of the layer of absorbent, 10 cm. The tests confirmed that chemical modification of lime chemical absorbents by water-soluble hydrophilic polymers, buffer solutions, and complexing agents is a promising method of increasing the absorbents' protective ability. When added to lime chemical absorbents, alkaline metal salts of polyacrylic acid increased the duration of the absorbents' protective effect with respect to CO_2 from 70 minutes (in the case of the nonmodified commercially available lime chemical absorbent) to between 95 and 96.9 minutes depending on the specific modifier used. This effect was linked to the increase in degree of neutralization of acid. The tests thus confirmed that chemical modification of lime chemical absorbents by water-soluble hydrophilic polymers, buffer solutions, and complexing agents is a promising method of increasing the absorbents' protective ability. The authors conclude by stating the need for more in-depth theoretical research on the characteristic features of the chemisorption processes occurring during such modification. Tables 4; references 13 (Russian).

Selecting Optimal Absorbents for Absorption Processes

927M0024H Leningrad ZHURNAL PRIKLADNOY KHMII in Russian Vol 64 No 3, Mar 91 (manuscript received 17 Oct 88) pp 582-589

[Article by V.N. Vetokhin, A.Yu. Komissarov, and Yu.V. Koritskiy]

UDC 66.02.06

[Abstract] The problem of selecting an effective absorbent must be addressed when developing and implementing processes of separating mixtures of gases with

close physicochemical properties. It is not currently possible to make a precise theoretical estimate of absorbents' effectiveness, and methods based on direct experimental research are laborious and often difficult to implement under laboratory conditions. For these reasons, the authors of the study reported herein worked to develop a theoretical method of finding effective absorbents. A series of 23 expressions for assessing an absorbent's effectiveness is presented along with the flowchart of an algorithm for computer-assisted comparative evaluation of several potential absorbents. The search for new absorbents is conducted in two stages. The first stage essentially entails the exhaustive trials of effective functional groups. The number of combinations checked in the second stage of the calculation is determined by the number of "effective" groups identified in the first stage. In the second stage, additional information about the given process or system is used to search among groups constituting a specified class of absorbents (e.g., aromatic hydrocarbons, alcohols, carbonyl compounds, etc.). Each potential absorbent in a given class is assessed both qualitatively and quantitatively based on its number of assigned points times weighting coefficients. The adsorbent with the highest weighting number of points is then considered the optimum absorbent in its class. The process is repeated until the optimum absorbent among all of the classes considered is found. Information about the optimum absorbent may then be used as a basis for synthesizing new absorbents. This is accomplished by searching for hypothetical substitute absorbents that would best meet the requirements for an absorbent in the specific case under consideration. This synthesis process may be implemented by conventional chemical methods under laboratory conditions or on a computer. The new absorbents are then subjected to tests on a pilot packed absorption unit. A comparison of data regarding a test absorbent that were obtained by (1) experimentation and (2) calculations based on the proposed model demonstrates that the using the new theoretical method of finding an effective absorbent can indeed produce satisfactory results. Figures 2, table 1; references 10: 5 Russian, 5 Western.

Determining Microquantities of Aluminum in Sewage by the Solid-Phase Spectrophotometry Method

927M0031C Moscow VESTNIK MOSKOVSKOGO UNIVERSITETA: KHIMIYA in Russian Vol 32 No 3, May-Jun 91 (manuscript received 6 Jun 90) pp 270-273

[Article by G.D. Brykina and I.P. Smirnov, Analytical Chemistry Department, Moscow University]

UDC 543.420.62:541.183.1

[Abstract] Because of the problems associated with using familiar reagents to determine microquantities of aluminum in complex objects, the authors of the study reported herein studied the determination of microquantities of aluminum in sewage by the solid-phase spectrophotometry method. Specifically, they investigated the properties of the anion exchanger AV-17x8 with grain sizes of 0.1-0.25 and 0.25-0.5 mm modified with anthrazochrome. During the course of their studies they refined several spectrophotometric characteristics of the complexing of aluminum with anthrazochrome in solution. The reagent and aluminum form a violet-colored complex compound in the pH range from 4.7 to 5.1. The color develops instantaneously and remains stable for several days. The maximum reagent absorption is observed at 505 nm, and the maximum complexing is observed at 580 nm. The molar coefficient of the absorption of the complex amounts to 2.4×10^4 . The studies also established that anthrazochrome containing two highly acidic sulfo groups is sorbed by the anion exchanger within a broad pH interval based on the ion exchange mechanism. The ion exchange sorption mechanism is complemented by the dispersion interaction of the π -electrons of the aromatic radicals of anthrazochrome with the ion exchanger's matrix. Complete absorption of the reagent under optimal conditions (pH 5-6) was accomplished within 30 minutes. A study of the light absorption of the reagent and its complexes with anthrazochrome both in solution and in the sorbent phase revealed that there is a regular shift in the maximum of the light absorption of anthrazochrome in the sorbent phase to the long-wave range when compared with the maximum light absorption of anthrazochrome in solution. The modified sorbent had a plane maximum light absorption at $\lambda = 540$ to 550 nm. The molar coefficient of the absorption of anthrazochrome in the sorbent phase amounted to $\epsilon_{540} = 2.1 \times 10^3$. The saturation curve method revealed that in the sorbent phase, a complex of aluminum and anthrazochrome forms with a 1:1 ratio and a value of ϵ_{540} amounting to 1×10^4 . Sorption of the complex obtained in a solution under optimum formation conditions at room temperature proved to occur easily because the complex is negatively charged. The threshold of aluminum detection amounted to 0.02 $\mu\text{g}/\text{ml}$. The method reported was used to analyze sewage from viscose fiber production at the Khimvolokno Production Association in Klin. The analysis results confirmed that the proposed method of solid-phase spectrophotometric determination of microquantities of aluminum in sewage is indeed feasible. Figures 2, tables 3; references 7 (Russian).

Effect of Liquid Crystal Molecular Structure on Their Physico-Mechanical Properties. Part I. Polar Derivatives of Pyridine

927M0021F Moscow *ZHURNAL FIZICHESKOY KHIMII* in Russian Vol 65, No 5, May 91 (manuscript received 15 May 90) pp 1356-1359

[Article by V. F. Petrov and M. F. Grebenkin]

UDC532.783

[Abstract] New chemical substances having liquid crystal properties continue to grow in number. Practical applications as components in liquid crystal materials hinge on the sum total of their physico-chemical properties, viz. phase transition temperature, optical, viscoelastic, and other characteristics. Thus, chemical structure and properties run hand in hand. However, a problem arises in that it is possible only to make an approximation of the properties of a liquid crystal from its structure, and even the approximation itself is frequently rests upon an analysis of the properties of a substance having a similar structure and utilizing a knowledge of the tendency in change in parameters of the substance with any specific change in its molecular structure. The present work was undertaken to search for correlations between molecular structural changes in liquid crystals and any corresponding changes in physico-chemical properties. Specifically, the effect that the type of polar terminal substituent, i.e., CN, -CH=CH-CN, NCS, F, has on the physico-chemical properties of liquid crystal derivatives of pyridine was studied. The data are presented in a table. References 12: 4 Russian, 8 Western.

Electrochemical Properties of Chemically Modified Diamond Surface

927M0021G Moscow *ZHURNAL FIZICHESKOY KHIMII* in Russian Vol 65, No 5, May 91 (manuscript received 13 Jun 90) pp 1364-1367

[Article by V. N. Deryabkin, A. N. Pushkin, V. V. Rassomakin, A. P. Rudenko, F. M. Taprayeva, and E. M. Trukhan, Moscow State University imeni M. V. Lomonosov]

UDC549.211+541.183.59+537.311

[Abstract] The presence of various atoms and functional groups on the surface of a solid body can alter its electronic state, and therefore have an effect on its surface conductivity. Thus, it has been established that the surface conductivities of germanium and silicon change considerably after adsorption with various gases. Chemosorption with gases associated with the surface formation of functional groups must also have an effect on the surface conductivity of semiconductors. Diamond is known to be a semiconductor material with a large forbidden zone width, and its surface may be characterized with various monofunctional coatings

(halogen, oxygen, hydrogen), depending on the conditions of modification. In the present work a study was made of the effect modification of synthetic diamond powder with hydrogen, methane, and air has on its surface, ultra-high frequency conductivity and Hall mobility of charge carriers. Diamond, treated with Moissan mixture (50 parts by volume 96 percent sulfuric acid, 30 parts by volume oleum, and 30 parts by volume 57 percent nitric acid), was demonstrated to have a specific surface conductivity of $0.7 \times 10^{-8} \text{ Ohm}^{-1}$. Modification of the same surface at low temperatures with hydrogen, methane, or air has no effect on specific conductivity. Figures 2; references 9: 8 Russian, 1 Western.

The Anodic Dissolution of Gold in Alkaline Cyanide Solutions. I. The Effect of Surface Development on Process Speed

927M0023A Novosibirsk *IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SIBIRSKIY KHIMICHESKIY ZHURNAL* in Russian No 3, May-Jun 91 (manuscript received 14 May 90) pp 30-35

[Article by R.Yu. Bek and G.V. Kosolapov, Solid-State Chemistry and Mineral Ore Refining Institute, Siberian Department, USSR Academy of Sciences, Novosibirsk]

UDC 541.135

[Abstract] In previous communications, the authors of the study reported herein demonstrated that heavy metal impurities and cyanide decomposition products are among the factors contributing to the qualitative and quantitative discrepancies among experimental data obtained by different authors regarding the process of the dissolution of gold in alkaline cyanide electrolytes. In the study reported herein, the authors continue their study of the said process. This time they focus their attention on the development of the metal surface during the dissolution process. As a working electrode, the authors used a torus of gold wire 0.5 mm in diameter that had been pressed into a Teflon sheathing. The electrode's surface was prepared by cutting a thin layer of metal from its surface with a ruby blade directly in the solution by using a device described elsewhere. A rotating disk electrode was used to study the anodic behavior of gold in a cyanide solution during agitation. A platinum wire was used as an auxiliary electrode, and a saturated silver chloride electrode served as the standard electrode. A JSM T-20 scanning electron microscope was used to perform the morphological studies of the electrode surface. The solutions used were prepared from bidistilled water and distillation-purified high-purity potassium cyanide. Before the experiments, oxygen was removed from the solution by blowing electrolytic hydrogen through it for 3 hours. During the experiments, the solution's temperature was kept at 25 or 50°C depending on the given experiment. The potentiostatic curves reflecting the gold's behavior after its surface was renewed at different potentials (from -0.5 to +0.6 V)

showed that the gold's behavior is indeed dependent on the potential. In the range of potential from -0.1 to +0.3 V the process achieves a steady state in about 20 minutes. In the case of more negative potentials, several hours are required to reach a steady state. A change in current over time is also observed. Next, a series of experiments examining surface morphology was performed to confirm that the observed change in current is connected with the change in the magnitude of the surface. This hypothesis was in fact confirmed to be valid throughout the entire range of potentials studied. The experiment results were interpreted as follows. Immediately after the surface is renewed, there is intensive dissolution of the gold from the microroughnesses formed as a result of the blade's movement. Over the course of time, the number of such active surface centers decreases. The rate at which the gold dissolves decreases as well (this is reflected in the descending leg of the current-time curve). The process then occurs on active centers of another type, for example, grain intergranular boundaries, and gradually results in an increase in the electrode's true surface. This in turn causes an increase in the process rate, which is reflected in the ascending leg of the current-time curve. In the case of potentials more positive than +0.3 V, no such strong surface development occurs. The studies reported thus confirmed that the effects of surface development occurring at potentials more negative than +0.3 V must be given consideration when researching the laws governing the anodic dissolution of gold. Figures 5; references 13: 6 Russian, 5 Western.

The Anodic Dissolution of Gold in Alkaline Cyanide Solutions. 2. The Effect of Alkaline Metal Cations

927M0023B Novosibirsk *IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SIBIRSKIY KHIMICHESKIY ZHURNAL* in Russian No 3, May-Jun 91 (manuscript received 2 Jul 90) pp 36-39

[Article by R.Yu. Bek and G.V. Kosolapov, Solid-State Chemistry and Mineral Ore Refining Institute, Siberian Department, USSR Academy of Sciences, Novosibirsk]

UDC 541.135

[Abstract] This article is the continuation of a series of studies devoted to the factors affecting the anodic dissolution of gold in alkaline cyanide solutions. In the present article, the authors focus their attention on the effect of the concentration and nature of alkaline metal cations on the said process. To prepare the study specimens, the authors used bidistilled water; high-purity lithium, sodium, and potassium hydroxides; high-purity potassium sulfate; high-purity potassium chloride; and potassium cyanide purified by a method described elsewhere. Before the experiments, oxygen was removed from the solution by blowing electrolytic hydrogen through for 3 hours. During the experiments, the solution's temperature was maintained at 25°C. Previous

research on the subject led the authors to expect that the effect of alkaline metal cations would be greatest at the maximally attainable current densities in the respective solutions, i.e., when the near-electrode concentration of $\text{Au}(\text{CN})_2^-$ is at its maximum. The potentiometric studies performed did not confirm this expectation. It turned out that increasing the concentration of potassium salts does not have a marked effect on the process rate in the region of the highest current densities, i.e., in the region of potentials around +0.6 V. On the contrary, the strongest depressing effect appeared in the range of potentials around +0.2 V, where the current density is far below the maximally attainable value. A comparison of the polarization curves of the dissolution of gold in alkaline cyanide solutions prepared on the basis of potassium, sodium, and lithium compounds led to an analogous conclusion: the nature of the cation has no effect in the range of potentials where the current densities of the gold's dissolution are at a maximum (around +0.6 V). Instead, they exert their strongest effect at more negative potentials, where the process rate is much slower. These findings indicate that the reigning interpretation in the literature, i.e., that the nature and concentration of alkaline cations affect the rate of gold dissolution, is erroneous. Figures 5; references 9: 6 Russian, 3 Western.

The Anodic Dissolution of Gold in Alkaline Cyanide Solutions. 3. The Effect of Hydroxide Ions

927M0023C Novosibirsk *IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SIBIRSKIY KHIMICHESKIY ZHURNAL* in Russian No 3, May-Jun 91 (manuscript received 5 Sep 90) pp 40-46

[Article by R.Yu. Bek and G.V. Kosolapov, Solid-State Chemistry and Mineral Ore Refining Institute, Siberian Department, USSR Academy of Sciences, Novosibirsk]

UDC 541.135

[Abstract] In a continuation of their previous research on the anodic dissolution of gold in alkaline cyanide solutions, the authors of the study reported herein examined the effect of hydroxide ions on the said process. As an electrode for their electronographic study of the metal's surface, they used a gold wafer measuring 20 x 8 x 3 mm that had been preliminarily etched in nitrohydrochloric acid and polished. The wafer was then placed into a cyanide electrolyte and held there for 30 minutes at the specified potential. It was then removed from the cell, rinsed with bidistilled water, dried, and used for the optronic measurements, which were taken by using an EF-4 optronic unit in transmission and reflection modes. Bidistilled water and high-purity starting substances were used for the experiments. The solution's temperature was kept at 25°C. Increasing the content of hydroxide ions in the case of potentials more positive than +0.3 V caused a shift in the descending arm of the first current peak (around +0.3 V) and in both arms of the second current peak (around +0.6 V) toward the

negative potential side. The first peak decreased in size, whereas the second remained virtually unchanged. At low pH (around 10-11) and high potassium cyanide concentrations (0.1 mol/l), the first peak approximated the second in size, and the drop after the peak was only weakly expressed. At low cyanide concentrations and high hydroxide concentrations, on the other hand, the first peak is absent. Passivation of the electrode as a result of adsorption of the hydroxide ions was deemed the likely cause of the drop in current at high pH after a potential of about +0.3 V was reached. Next, the researchers turned their attention to the reason for the new increase in current observed at potentials more positive than +0.4 V and resulting in the appearance of yet another maximum in the vicinity of +0.6 V. Morphological studies revealed that etching pits appear on the electrode's surface at potentials more positive than +0.4 V. They were associated with breaks in the continuity of the passivating film caused by its reconstruction prior to the formation of a phase of trivalent gold oxide that forms at potentials more positive than +0.6 V. The number of these pits were found to reach a maximum in the region of +0.6 V and then falls off. The authors hypothesized that the dissolution process is localized at these sites. The authors also postulated the following law governing the effect of the relationship of the cyanide and hydroxide concentrations on the gold surface at potentials between +0.3 and +0.5: When the hydroxide concentration remains constant, the current increases as the cyanide concentration increases; when the cyanide concentration remains constant, the current decreases as the hydroxide concentration increases. Figures 6, table 1; references 20: 15 Russian, 5 Western.

The Solubility of AgCl in Aqueous Solutions of HCl and HAuCl₄

927M0023E Novosibirsk IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SIBIRSKIY KHMICHESKIY ZHURNAL in Russian No 3, May-Jun 91 (manuscript received 30 Jan 90) pp 62-66

[Article by V.I. Belevantsev and L.D. Tsvelodub, Inorganic Chemistry Institute, Siberian Department, USSR Academy of Sciences, Novosibirsk]

UDC 541.49'123.22+546.57'59'131

[Abstract] The authors of the study reported herein studied the solubility of AgCl in aqueous solutions of HCl and HAuCl₄ at temperatures of 16, 42, and 62°C. The relative concentrations of HCl and HAuCl₄ were varied in three series of three experiments each. In the first series the concentration of HAuCl₄ was kept at 1.54 mol/l for the three experiments while three different concentrations of HCl were used (1.00, 2.00, and 2.99 mol/l). In series 2, the HAuCl₄ concentration was kept at 0.925 mol/l, and HCl concentrations of .099, 1.99, and 2.92 mol/l were used. In series 3, HCl concentrations of 1.00, 2.02, and 3.00 mol/l were used along with an HAuCl₄ concentration of 0.610 mol/l in each case.

Analysis of the experiment results revealed that both the makeup of the electrolyte and the reaction temperature exerted significant and regular effects on the solubility of AgCl. They offer the following model of the solubility of AgCl in aqueous solutions of HCl and HAuCl₄:

$$C_{Ag} = K_1(I)[Cl^-] + K_2(I)C_{Au}/[Cl^-]$$

Through a series of computations, they demonstrate that the model provides a good description of all of the experimental data obtained. The authors further concluded that their experiments make it possible to reject the model assuming a broader range of the existence of the two complexes AgCl₂⁻ and AgCl₃²⁻ without any significant contribution by AuCl₃AgCl. They conclude by stating that the role of the complex AgCl₂⁻ is decidedly most significant in the solubility of AgCl under the conditions studied herein. Figure 1, tables 5; references 3: 1 Russian, 2 Western.

A Study of the Sorption Properties of Selective Ion Exchangers by Atomic Absorption Spectrometry With a Laser Atomizer

927M0023F Novosibirsk IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SIBIRSKIY KHMICHESKIY ZHURNAL in Russian No 3, May-Jun 91 (manuscript received 30 Nov 90) pp 95-100

[Article by Ye.K. Vulfson, Ye.G. Abramov, E.M. Kats, and I.B. Serova, Geochemistry and Analytical Chemistry Institute imeni V.I. Vernadskiy, Moscow]

UDC 541.183:543.42

[Abstract] The authors of the study reported herein investigated the sorption properties of a number of selective ion exchangers with various commercially, semicommercially, and laboratory-manufactured functional groups. Four types of ion exchangers were examined: anion exchangers (An-31, AN-22, ANG-11g, AM-7, AN-511, AMOKH, SB-1 PEPA, and SB-1 [shop-produced]); cation exchangers (KF-7, KF-11, and KB-4); amphotolytes (ANKB-50, ANKB-35, ZAK-3, AMK-1, PA-12, and GIPO); and fiber ion exchangers (VION KN-1, Mtilon-T [nonreinforced], and Mtilon-T [reinforced]). The sorbent studies were performed in model solutions (pH, 2 to 8) and in natural sea water. The sorption of copper, nickel, zinc, cobalt, and manganese in solutions containing the specified metals in the concentration range from 1 mg/l to 3-5 µg/l was studied. The process of the sorption and desorption of the metals was controlled by determining their content by atomic adsorption spectrometry with flame atomization as well as by direct determination of the elements in the ion exchanger phase by atomic absorption spectrometry with a laser atomizer. Most of the anion and cation exchangers studied were found to contain insignificant amounts of microcomponents (µg/g): copper, 0.5 to 4; nickel, 0.5 to 0.8; zinc, about 2; and cobalt, <0.5. The only exception was the shop-produced batch of SB-1, which contained somewhat more copper and zinc. Some of the

ampholites and fiber ion exchangers (AMK-1, GIPO, VION KN-1) contained significant amounts of copper (125 to 225 $\mu\text{g/g}$). The following were singled out as promising ion exchangers for extracting Cu, Ni, Zn, and Co from sea water: AN-31, AM-7, AMOKH, CB-1, PA-12, KB-4, VION KN-1, and nonreinforced Mitolon-T. Data from full-scale tests of each of these ion exchangers in either sea water or simulated ocean water regarding their absorption of the said metals are presented in table form. The fiber ion exchangers were found to have a higher operating efficiency in a unit with a stationary layer of ion exchanger than in a submersible unit. Tests conducted on the selective hydroxamino ion exchanger SB-1 revealed a significant concentration of the study microcomponents. The degree of copper extraction by SB-1 was found to be 80% or more. The amount of metal absorbed by the SB-1 in 11 cycles was eight- to tenfold more than in one cycle and amounted to 10^3 mg/l or more. Further studies revealed that complete desorption of nonferrous metals from SB-1 is possible by using diluted solutions of mineral acids. Figure 1, tables 4; references 4 (Russian).

The Behavior of Yttrium and Barium Cuprates at High Temperature in the System $\text{Y}_2\text{O}_3\text{-BaO-CuO}$

927M0024K Leningrad ZHURNAL PRIKLADNOY KHMII in Russian Vol 64 No 3, Mar 91 (manuscript received 7 May 90) pp 662-665

[Article by O.G. Chigareva, G.A. Mikirticheva, V.I. Shitova, L.Yu. Grabovenko, S.K. Kuchayeva, and R.G. Grebenshchikov, Silicate Chemistry Institute imeni I.V. Grebenshchikov, USSR Academy of Sciences]

UDC 537.312.62:541.124.16:541.118:546.562:546.431

[Abstract] In a study of the phase relationships existing in particular sections of the pseudo-ternary system $\text{Y}_2\text{O}_3\text{-BaO-CuO}(\text{Cu}_2\text{O})$ the authors established that the temperature bounds of the phase fields plotted based on derivative thermogravimetric analysis data were 30 to 80° higher than those plotted on the basis of data obtained during extended annealing of specimens. To explain this discrepancy, the authors of this concise report conducted a study to determine the thermal stability of the compounds Y_2BaCuO_5 (the "green phase"), $\text{Y}_2\text{Cu}_2\text{O}_5$ (the "blue phase"), BaCuO_2 , and $\text{YBa}_2\text{Cu}_3\text{O}_{7-8}$ (1:2:3) crystallized in the aforesaid system. All four compounds were synthesized in air by the solid-phase method. Monolithic pieces of the specimens were then placed in a platinum crucible and annealed for between 0.5 and 6 hours, after which they were quenched in ice or liquid nitrogen. The temperature of the initial annealing for all compounds was 950°. Each subsequent annealing was 30° higher than the previous one, and the annealing process was repeated until each individual specimen melted. The phase composition of identical specimens was established based on x-ray and microscopy analyses. The curve plotted from derivative thermogravimetric analysis of $\text{Y}_2\text{Cu}_2\text{O}_5$ reveals three

endothermal effects. The one in the interval from 1,150 to 1,190°C corresponds to a decrease in specimen mass. This main endothermal effect (with its maximum at 1,190°C) is tied to the incongruent melting of the blue phase into Y_2O_3 and liquid. Protracted annealings of the blue phase in the temperature interval from 1,050 to 1,180°C in air demonstrated that it dissociates at a lower temperature. The large endothermal effect that reaches its maximum at 1,310°C corresponds to a mass loss on the thermogravimetric curve. This effect is tied to the incongruent melting of the green phase. Upon protracted heating in air in the temperature interval from 1,220 to 1,280°C, Y_2BaCuO_5 is, like the blue phase, subject to phase changes expressed in the "lightening" of the specimens and the partial dissociation of the green phase into Y_2O_3 and a melt. A specimen of Y_2BaCuO_5 annealed for 6 hours at 1,200°C was found to maintain its green color and remain unchanged according to x-ray studies. The authors also conducted tests to determine the crystallization temperature of the blue and green phases about 100° higher than their temperatures of incongruent melting, i.e., to 1,400 and 1,300°, respectively. Crystallization of the blue and green phases was found to occur at temperatures below 1,070 and 1,220°, respectively. It was further discovered that within the makeup of its blue phase, copper is preserved in the form of Cu (II) upon heating to 1,070°C, whereas in Y_2BaCuO_5 it remains stable to 1,220°C. This is significantly higher than in copper (II) oxide (which remains stable until about 1,030°C). Studies of the thermal behavior of the aforesaid yttrium and barium cuprates in air demonstrated that both have a low thermal stability and that their stability is a function of the dynamics of their heating. In order of decreasing temperature of decomposition, the said compounds may be ranked as follows: Y_2BaCuO_5 — $\text{Y}_2\text{Cu}_2\text{O}_5$ — CuO — BaCuO_2 — $\text{YBa}_2\text{Cu}_3\text{O}_{7-8}$. Figure 1; references 5: 4 Russian, 1 Western.

The Reaction of Halfnium-Scandium Alloys With Hydrogen

927M0025D Moscow ZHURNAL NEORGANICHESKOY KHMII in Russian Vol 36 No 6, Jun 91 (manuscript received 22 Oct 90) pp 1377-1379

[Article by V.N. Verbetskiy and E.A. Movlaiyev, Moscow State University imeni M.V. Lomonosov]

UDC 546.121

[Abstract] The authors of this study examined the reaction of halfnium-scandium alloys with hydrogen. A total of 13 different alloys produced from scandium with a purity of 99.87% and from halfnium with a purity of 99.9% were subjected to hydrogenation at a pressure of 15 to 25 atm. The alloys and hydride phases were then subjected to x-ray phase analysis on a DRON-2 diffractometer using CuK_{α} -radiation. The periods of the lattice were determined with a precision of $\pm 0.05\%$. The

starting specimens were single-phase alloys with a hexagonal lattice. The alloys' lattice periods were found to decrease slightly as the halfnium content was increased. This was due to the slight difference between the atomic radii of Hf and Sc. No noticeable gas absorption was observed when the alloys were reacted with hydrogen at room temperature. Upon heating to 200-250°C, the reaction between the components began immediately without any preliminary activation or induction period and finished after 30 to 40 minutes. As the scandium content of the study alloys increased, the temperature at which the alloys began to react with the hydrogen was found to decrease slightly (from 250 to 200°C). The amount of gas absorbed increased simultaneously. The hydrides with more than a 70 atomic percent halfnium content were found to crystallize in a tetragonal lattice analogous to halfnium dihydride. The transition in the said hydrides from a face-centered crystalline lattice to a tetragonal lattice was discovered to occur in a very narrow concentration interval, i.e., when the scandium content ranged from 35 to 30 atomic percent. Differential thermal analysis revealed that not one of the hydrides produced evolves hydrogen. The results obtained confirmed the authors' initial hypothesis of the mutual solubility of halfnium and scandium hydrides. The dihydride phase will, depending on the ratio of its metal components, be either a scandium hydride or a halfnium hydride. The range of the existence of an ϵ -phase in the system Hf-Sc-H₂ is much broader than in the system Zr-Sc-H₂ (30 atomic percent in the former case versus 10 atomic percent in the latter). Table 1; references 6 (Russian).

Binary Selenates of Rare Earth Elements and Ammonium

927M0025E Moscow ZHURNAL NEORGANICHESKOY KHIMII in Russian Vol 36 No 6, Jun 91 (manuscript received 15 Nov 90) pp 1380-1381

[Article by L.D. Iskhakova, N.P. Kozlova, and L.G. Makarevich]

UDC 546.23'65'39

[Abstract] The authors of this concise report worked to establish both the compositions of NH₄Ln(SeO₄)₂·nH₂O and the structural types in which they are crystallized. The said crystalline hydrates were extracted during the process of isothermal evaporation (at 20-25°C) of solutions containing equimolar quantities of selenates of ammonium and the respective rare earth metal. Selenic acid was added until a pH of 2-3 was achieved. NH₄Ln(SeO₄)₂ compounds were produced by high-temperature recrystallization of the crystalline hydrates in a 20% solution in a 20% solution of selenic acid at a temperature of 200 to 220°C for 24-36 hours. The resultant compounds were subjected to x-ray crystallographic studies. None of the roentgenograms of any of

the specimens x-rayed contained additional reflexes corresponding to any impurity phases. The fact that all of the compounds produced were single-phase compounds confirmed the congruent nature of their dissolution. From the standpoint of composition, the binary selenates synthesized turned out to be analogues of the crystalline hydrates MLn(SeO₄)₂·nH₂O with potassium and rubidium. The authors were only able to succeed in obtaining anhydrous NH₄Ln(SeO₄)₂ compounds for Ln = La, Pr-Sm. In the case of Ln = Ce, a Ce^{III} → Ce^{IV} oxidation process occurred that resulted in the formation of slightly soluble simple cerium (IV) selenates. All of the NH₄Ln(SeO₄)₂ synthesized was isostructural to MLn(SeO₄)₂ selenates with M = K, Rb, and Cs and Ln = La-Nd and belonged to the monoclinic structural type KNd(SO₄)₂. Table 1; references (Russian).

The Crystalline Structure of Bromine Trifluoride at 120°C

927M0025F Moscow ZHURNAL NEORGANICHESKOY KHIMII in Russian Vol 36 No 6, Jun 91 (manuscript received 7 Dec 90) pp 1393-1396

[Article by A.M. Ellern, M.Yu. Antipin, Yu.T. Struchkov, and V.F. Sukhoverkhov, Elementoorganic Compounds Institute imeni A.N. Nesmeyanov, USSR Academy of Sciences, and General and Inorganic Chemistry Institute imeni N.S. Kurnakov, USSR Academy of Sciences]

UDC 548.734

[Abstract] The authors of this report conducted an x-ray crystallographic study of BrF₃ in order to determine its molecular and crystalline structure. The BrF₃ was synthesized and purified in accordance with the familiar method. All of the operations entailed in purifying the substance and filling and sealing off the capillaries were performed in a vacuum system of Pyrex glass. The monocrystals were grown directly on a Sinteks P2₁ diffractometer with a low-temperature attachment by the Bridgman method. The crystals were slowly cooled to a temperature of 120°C, at which point they were subjected to an x-ray diffraction experiment. The BrF₃ crystals produced were rhombic and had parameters close to those found previously at 120°C: a = 5.315(1), b = 7.305(1), and c = 6.624(1) angstroms, V = 257.18(7) cubic angstroms; Z = 4; d = 3.536 g/cm³; and limiting boundary, Cmc2₁. The study results indicated that, in complete accordance with Gillespie's theory, at 120°C, BrF₃ molecules are plane with three nonequivalent fluorine atoms. Only one of the axial F atoms of each molecule participates in the additional coordination of the adjacent Br atoms. The molecules in the crystal were found to be arranged in layers parallel to the yz plane, and chains were found to form in each layer owing to the additional coordination Br...F(3'). The nonvalence distance Br...F(3') was found to equal 2.45 angstroms, which is much less than the sum of the Van der Waals

radii for these atoms (i.e., 3.37 angstroms). The authors conclude by noting that the nature of the packing of crystalline BrF_3 is close to the packing in the low-temperature modification of ClF_3 . Figure 1, table 1; references 13: 9 Russian, 4 Western.

The Crystalline Structure of $\text{Ba}_7\text{Sc}_6\text{Al}_2\text{O}_{19}$

927M0025H Moscow ZHURNAL NEORGANICHESKOY KHIMII in Russian Vol 36 No 6, Jun 91 (manuscript received 25 Oct 90) pp 1402-1407

[Article by R.V. Shpanchenko, Ye.V. Antipov, M.V. Paromova, and L.M. Kovba, Moscow State University imeni M.V. Lomonosov]

UDC 548.736.5

[Abstract] The authors of the study reported herein examine the crystalline structure of $\text{Ba}_7\text{Sc}_6\text{Al}_2\text{O}_{19}$ synthesized in air at 1,500°C for 24 hours with intermediate wiping. X-ray crystallography studies of powder specimens revealed that the structure of $\text{Ba}_7\text{Sc}_6\text{Al}_2\text{O}_{19}$ is a 14-layer packing of BaO_{3-x} layers. The oxide $\text{Ba}_7\text{Sc}_6\text{Al}_2\text{O}_{19}$ was found to be an unusual example of perovskite-like compounds of the composition $\text{A}_n\text{B}_{n+1}\text{X}_{3n-x}$. The authors state that it is only the second example (after $\text{Ba}_3\text{R}_4\text{O}_9$) of the formation of hexagonal perovskite-like phases with passage of the AX_3 layers into a derivative of 16-layer very dense packing. They further state that $\text{Ba}_7\text{Sc}_6\text{Al}_2\text{O}_{19}$ is the first compound where the passage of the layers into a very dense cationic-anionic packing is accompanied by ordering of the anionic vacancies. The structure of $\text{Ba}_7\text{Sc}_6\text{Al}_2\text{O}_{19}$ may, according to the authors, be looked upon as a structure of the intergrowth of fragments of $\text{Ba}_3\text{Sc}_4\text{O}_9$ and $\beta\text{-Ba}_2\text{ScAlO}_3$ structures along the c axis. This fact leads the authors to hypothesize that similar structures may form during the intergrowth of fragments of very dense packings or blocks with different cationic and anionic makeups. This would in turn permit the purposive synthesis of new hexagonal perovskite analogues. Figures 2, tables 3; references 7: 5 Russian, 2 Western.

Extraction of Thallium (I) by Selected Crown Ethers

927M0031B Moscow VESTNIK MOSKOVSKOGO UNIVERSITETA: KHIMIYA in Russian Vol 32 No 3, May-Jun 91 (manuscript received 6 Jun 90) pp 258-261

[Article by Kim Yen Gvan and B.Z. Iofa, Radiochemistry Department, Moscow University]

UDC 542.61:546.683

[Abstract] Previous research has shown that the logarithm of the extraction constant of 15-crown-5 sodium picrate decreases linearly as the inverse of the dielectric constant of the diluent increases. The authors of the study reported herein attempted to use the existing

literature data to find a similar law based on the example of the extraction of thallium (I) by crown ethers. They determined that the values presented in the literature for the extraction constants of thallium (I) picrate by chloroform solutions of crown ethers are somewhat low. For that reason, they studied the extraction of thallium (I) picrate by chloroform solutions of 15-crown-5, 18-crown-6, dibenzo-18-crown-6, and dinitrobenzo-18-crown-6. They determined the coefficients of the distribution of thallium (I) picrate in the presence of crown ethers between water and chloroform radiometrically by means of the β -radiation of thallium 204 ($T_{1/2} = 3.81$ years, $E\beta = 0.766$ meV). They found that the logarithms of the extraction constants of thallium (I) picrate are linearly dependent on the inverse of the dielectric constant of the diluents studied. They conclude by emphasizing that as in the case of extraction of 15-crown-5 sodium picrate, the free energy of the extraction of thallium (I) picrate may be broken down into the sum of two terms, with the second term characterizing the purely physical (electrostatic) interaction in the extracted ion pair. The log of the extraction constant is discovered to be a linear function of the inverse of the dielectric constant of the crown ether diluent. It is hypothesized that a similar pattern occurs for other elements as well. Figures 2, table 1; references 13: 2 Russian, 11 Western.

The Synthesis of Functional Macroyclic Nickel Complexes and Their Immobilization on a Disperse Silica Surface

927M0040A Kiev UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 10, Oct 91 (manuscript received 18 Dec 90) pp 1038-1042

[Article by S.S. Silchenko and A.G. Kolchinskiy, Surface Chemistry Institute, UkrSSR Academy of Sciences, Kiev]

UDC 543.544.2

[Abstract] The primary objective of the study reported herein was to immobilize aminomethyl- and carboxymethyl-substituted macrocyclic nickel complexes on a disperse silica surface. Because grafting silica at silanol groups results in the formation of hydrolytically unstable SiOC and SiNC bonds, specimens of silica with graft propylamino or propylcarboxy groups were used. Carboxypropyl silica gel was produced by modifying Silpearl silica gel by γ -cyanopropylmethyldichlorosilane in toluene followed by hydrolysis of the CN groups by a mixture of diluted acetic and sulfuric acids. The concentration of carboxyl groups on the surface was determined to be 1.7 mmol/g (2.83 $\mu\text{mol}/\text{m}^2$). Aminopropyl silica gel was produced by liquid-phase modification of L100/250 and Silpearl silica gels by γ -aminopropyltriethoxy silanol in an aqueous solution at 60°C for 1 hour. The concentration of amino groups on the L100/250 silica gel was determined to be 0.58 mmol/g (0.97 $\mu\text{mol}/\text{m}^2$), and that on Silpearl was determined to be 0.43 mmol/g (0.71 $\mu\text{mol}/\text{m}^2$). Two versions of the same basic technique

were used to immobilize aminomethyl-substituted macrocyclic complexes. Both techniques, as well as the method used to immobilize carboxymethyl-substituted macrocyclic nickel complexes, involved the formation of peptide bonds. Carbodiimide and acid chloride were used as activators. Specimens obtained under soft conditions (the carbodiimide method) maintained their lilac coloring in a weakly alkaline medium. Upon acidification by acetic acid, their color changed to yellow. This change was demonstrated to be partially reversible upon alkalization by ammonia. When the specimens were dried or else produced under stricter conditions (the acid chloride method), their yellow coloring did not change upon alkalization. This behavior was explained in terms of the formation of a high-spin macrocyclic nickel complex with axially coordinated OH^- ions in an alkaline medium versus the formation of a low-spin square-planar complex in an acidic medium. When the specimens are stored or dried, there is a strong irreversible sorption of the macrocyclic part of the graft complex due to the formation of hydrogen bonds between the nitrogen atoms and silanol groups. The graft complexes hold fast to the silica gel surface and are not even washed off upon treatment with a 10% sulfuric acid solution. The graft complexes may, however, be removed from the surface by combined treatment with a strong oxidizing agent (such as sodium persulfate) and acid. Figures 2; references 9: 4 Russian, 5 Western.

The Anionic Structure of Glasses of the System (1-x-y) NaPO_3 : $x\text{NaF}$: $y\text{LiF}$

927M0040B Kiev UKRAINSKIY KHIMICHESKIY
ZHURNAL in Russian Vol 57 No 10, Oct 91
(manuscript received 7 Mar 90; after revision 27 Jun 91)
pp 1043-1046

[Article by A.A. Pronkin, N.I. Yumashev, A.A. Ilin, and
L.V. Yumasheva, Leningrad Technological Institute]

UDC 548.219.3:546.185

[Abstract] The authors of the study reported herein examined aqueous solutions of glasses of the system (1-x-y) NaPO_3 : $x\text{NaF}$: $y\text{LiF}$ (where $x = 0$ and $y = 0$ to 0.3 and where $y = 0$ and $x = 0.3$). The glasses were synthesized in crucibles of type CU-2000 glassy carbon in an argon medium over the course of 20 minutes at a temperature of 800°C. The charges were prepared from chemically pure NaPO_3 , LiF , and NaF . The melt was poured into heated metal forms, and the specimens were roasted at a temperature between 250 and 300°C for 4 hours. The specimens were subjected to ^{31}P and ^{19}F NMR spectroscopy and secondary-ion ionization mass spectrometry studies. The studies established eight types of fluorophosphate anions containing fluorine atoms at the ends of the polyphosphate chains and fluoride anions. After analyzing the ^{19}F and ^{31}P NMR spectra of the study system, the researchers classified the signals observed as specific phosphate-fluoride groupings in which the fluorine atoms are covalently bound with the

phosphorus atoms and are located at the ends of the polyphosphate chain. A total of eight types of fluorophosphate anions were established: $[\text{PO}_3\text{F}]^{2-}$, $[\text{P}_2\text{O}_6\text{F}]^{3-}$, $[\text{PO}_2\text{F}_2]^-$, $[\text{P}_2\text{O}_5\text{F}_2]^{2-}$, $[\text{P}_3\text{O}_8\text{F}_2]^{3-}$, $[\text{P}_4\text{O}_{11}\text{F}_2]^{4-}$, $[\text{P}_5\text{O}_{14}\text{F}_2]^{5-}$, and $[\text{P}_6\text{O}_{17}\text{F}_2]^{6-}$. The values of the chemical shifts of the ^{19}F and ^{31}P nuclei and constants of the spin-spin interactions forming the P-F bond in the specimens were determined. The authors also established the quantitative distribution of fluorine atoms with respect to the corresponding anions as a function of the concentration of MeF (where $\text{Me} = \text{Na}$ and Li) added: The addition of the said alkaline metal fluorides to the study glasses was manifested primarily in the breaking of the polyphosphate chain and a reduction in the fraction of phosphorus atoms in the long-chain phosphates from 0.91 to 0.47 (for 30 mol% MeF) and in the formation of mono- and difluoride oligomers. Cyclic tri- and trimetaphosphate anions were found to be stable in relation to the action of MeF . The studies performed also established that the equimolar substitution of 0.3 NaF by 0.3 LiF results in the following differences in the phosphate component of the study glasses: 1) the relative fraction of phosphorus atoms in $[\text{PO}_3\text{F}]^{2-}$ and $[\text{P}_2\text{O}_6\text{F}]^{3-}$ decreases by a factor of 2 and 1.5, respectively; 2) tripolyphosphate anions (which are absent in glasses of the composition $(1-x)\text{NaPO}_3$: $x\text{NaF}$) are observed to form; and 3) the relative fraction of phosphorus atoms in the tripolyphosphate anions amounts to 0.016, and the concentration of trimetaphosphate anions is twice as high. Figures 2; references 6: 2 Russian, 4 Western.

The Thermodynamic Properties of Chromium Borides in a Broad Temperature Interval

927M0040C Kiev UKRAINSKIY KHIMICHESKIY
ZHURNAL in Russian Vol 57 No 10, Oct 91
(manuscript received 3 Dec 90) pp 1060-1064

[Article by A.S. Bolgar, A.V. Blinder, and M.I. Serbova,
Materials Science Problems Institute, UkrSSR Academy
of Sciences]

UDC 536.722+546.271

[Abstract] The authors of this article conducted an experimental study of the heat capacity and enthalpy of chromium mono- and diboride in the temperature interval extending from 150 to 2,200 K. They also calculated the parameters of the temperature dependences of the thermodynamic functions of the study compounds in the interval from 298.15 to 2,200 K. The study compounds were produced by synthesis from the individual elements and subsequent annealing in a vacuum. The study CrB_2 contained the following (% by mass): Cr, 70.6; B, 29.3; O, 0.03; C, 0.03; and Fe, 0.05. The study CrB contained the following (% by mass): Cr, 82.6; B, 17.2; O, 0.02; C, 0.04; and Fe, 0.11. The dynamic calorimeter method was used to measure the heat capacity of the study chromium borides at temperatures between 150 and 675 K (with a measurement error not exceeding 4%). The enthalpies determined for

CrB and CrB_2 in the present study were found to be in satisfactory (within 3%) agreement with previously reported values, and the discrepancy between the heat capacity values reported herein and elsewhere reached 10% at 1,000 K. This discrepancy was attributed to the imprecision of the approximating polynomial used elsewhere. The study CrB was determined to have Debye and Einstein temperatures of 587 K and 981 K, respectively. The Debye and Einstein temperatures of the study CrB_2 were determined to be 265 and 851 (864), respectively. The authors concluded that in the high-temperature range, the heat capacity of the study compounds may be represented as the sum of the electronic and harmonic portions of the lattice component and that, in the case of monoboride, it may also be represented in terms of the contribution of anharmonism to the vibrations of the atoms of the crystalline lattice. Tables 2; references 10: 8 Russian, 2 Western.

Protolytic Properties of Silica Sorbents

927M0040D Kiev UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 10, Oct 91 (manuscript received 11 Oct 90) pp 1073-1076

[Article by A.N. Chebotarev, V.G. Markova, and Nguen Dang Dyk, Odessa University]

UDC 541.452/454:456.284-31

[Abstract] The authors of this article used the method of pH-metric heterogeneous titration to study the protolytic properties of a series of amorphous silica sorbents that are distinguished by their dispersivity and by the structural features of their surfaces. Specifically, types A-175, A-300, and A-380 aerosol were studied along with type K-7-30 dimethyl aerosil. L 100/250 macroporous silica gel was also studied for the sake of comparison. The kinetics of the changes in the pH of aqueous suspensions of the said silicas were studied, and the isolectric point of each of the silicas was determined by two independent methods (i.e., by microelectrophoresis and the drift method). The isolectric point of all of the study aerosols and the study silica gel were in the pH interval from 2.5 to 3. In the pH interval from 0 to 2.5 or 3 the silica's surface is positively charged due to protonation of the silanol groups. At higher pH values, the surface of the silicas is negatively charged due to their partial dissociation. The dissociation constants of each of the study compounds was determined and summarized in table form. The K-7-30 modified dimethyl aerosil was determined to be more acidic than the others, although its concentration of residual silanol groups is about an order of magnitude below than of the remaining aerosils. This is explained by the fact that most of the surface silanol groups of the nonmodified aerosils are involved in a complex system of hydrogen bonds by adsorbed water molecules, which greatly interferes with the dissociation of the SiOH groups. In the case of modified K-7-30 aerosil, the residual SiOH surface groups are isolated from involvement in the continuous network of hydrogen bonds

by hydrophobic methyl groups, which in turn facilitates an increase in the lability of the proton of the silanol groups and an intensification of their acidity. The studies performed established that the protolytic properties of A-175, A-300, and A-380 aerosols is virtually independent of their dispersivity. The acid properties of the study silicas were markedly affected by the structural distinctions of their surface. L 100/250 macroporous silica gel was found to be practically an order of magnitude less acidic than non-porous silicas are. Figures 2, table 1; references 11: 5 Russian, 6 Western.

The Effect of γ -Irradiation on the Low-Temperature Reduction of Co_3O_4 by Hydrogen

927M0040E Kiev UKRAINSKIY KHIMICHESKIY ZHURNAL in Russian Vol 57 No 10, Oct 91 (manuscript received 16 Nov 90) pp 1084-1087

[Article by V.M. Belousov, L.V. Lyashenko, I.V. Bacherekova, E.V. Rozhkova, and Ye. Stokh, Physical Chemistry Institute, UkrSSR Academy of Sciences, Kiev]

UDC 539.2+541.183.5

[Abstract] The authors of this article studied the effect that preliminary γ -irradiation of Co_3O_4 has on its reaction with hydrogen at temperatures between 77 and 320 K. Cobalt (II) and (III) oxides were produced in accordance with the familiar method. The speed of the reaction of Co_3O_4 with hydrogen was measured in a static vacuum unit (described elsewhere) under a hydrogen pressure of 1.3 to 100 Pa. The x-ray photoelectron spectroscopy method was used to study the effect of γ -irradiation on the state of the cobalt and oxygen ions on the oxide surface. A VG-ESCA-3 photoelectron spectrometer was used. The presence of two types of oxygen ions was established: lattice ions with a bond energy of 529.8 eV and surface ions with a bond energy of 531.7 eV. The ratio of surface to lattice oxygen ions decreased significantly in the irradiated specimen. This was attributed to dehydration of the surface of the Co_3O_4 upon γ -irradiation, as is the case with other semiconductors. The researchers reasoned that because the $\text{Co}^{3+}/\text{Co}^{2+}$ ratio remains unchanged upon irradiation, it is likely that the freed electrons are captured by the oxygen vacancies, thus forming surface S centers. This results in the creation of new additional adsorption centers for hydrogen that in turn result in an increase in the reaction rate constant and a decrease in the process activation energy. Dehydration of the surface was determined to not only create new adsorption centers but also reduce the surface barrier, thus making it easier for an electron to tunnel from the hydrogen on the surface to the cobalt ions in the near-surface oxide layer. A rather strong aftereffect was observed in the study specimen: The elevated reduction rate was found to persist for more than 3 months. This was seen as confirmation of the fact that the electron capture occurs in deep traps. The study thus confirmed the previously proposed tunnel mechanism of the reduction of Co_3O_4 at low temperatures. Figures 1, tables 2; references 5 (Russian).

Active Sites on Surface of Superconductor Ceramic $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$

927M0021B Moscow *ZHURNAL FIZICHESKOY KHIMII* in Russian Vol 65, No 5, May 91 (manuscript received 18 Jun 90) pp 1214-1220

[Article by D. M. Sakov, A. G. Dipson, Ye. I. Saunin, V. A. Kuznetsov, V. V. Gromov, and Yu. P. Toporov, Physical Chemistry Institute, Moscow]

UDC538.945

[Abstract] Although studies of the surface and composition of the title ceramic following ionic etching showed significant stoichiometric deviation in surface composition, little attention has been devoted to the physical chemical processes taking place on the activated surface. Furthermore, the actual surface properties of this ceramic, after being subjected to mechanical action or radiation, must depend heavily on the presence of electron or hole active sites. It is feasible that these sites could participate in the migration of oxygen and in the interactions of the superconductor surface with the medium. In the present work heat stimulated exoemission and electron paramagnetic resonance were used to study the active sites formed on the surface of the title ceramic as a consequence of mechanical agitation or γ -radiation. It was established that these sites consist of Cu^+ ions bound to oxygen vacancies that are stable at or above 400 K, as well as the formation of Cu^{+3} and O^{2-} , which are stable at or below 550 K, but decompose at higher temperatures in the presence of neutral oxygen. Figures 5; references 10: 7 Russian, 3 Western.

Effect of Metal-Semiconductor Phase Transition on Surface Energy of Vanadium Dioxide

927M0021E Moscow *ZHURNAL FIZICHESKOY KHIMII* in Russian Vol 65, No 5, May 91 (manuscript received 20 Dec 89) pp 1354-1356

[Article by V. I. Miller, V. P. Demidov, N. N. Bulgakov, Omsk Polytechnical Institute]

UDC539.211:539.87

[Abstract] A Semiconductor-metal phase transition, taking place at a known temperature, makes this a desireable property in electronic devices. Best known is vanadium dioxide, which has a phase transition at 338.7 K. Unfortunately, single crystals of this substance are not very rigid and disintegrate after repeated transitions, thereby severely limiting their application. Monoclinic distortion in the vanadium dioxide crystal lattice is known to take place during phase transition, although how the surface energy, which determines the rigidity of solids, changes, is still not clear. In the present work the surface energies of ideal vanadium dioxide crystal facets were calculated for both the "metal" and "semiconductor" phases. It was demonstrated that surface energy diminishes during transition from the "semiconductor"

to the "metal" phase, and that of the surface facets forming a real vanadium dioxide crystal, the (100) and (010) are the most stable. The disintegration of a vanadium dioxide single crystal during phase transition is explained on the basis of the derived surface energy values. References 8: 7 Russian, 1 Western.

Hydrogen Stimulated Impregnation of Contaminants Through Palladium Alloy Membranes

927M0021J Moscow *ZHURNAL FIZICHESKOY KHIMII* in Russian Vol 65, No 5, May 91 (manuscript received 3 Aug 90) pp 1419-1421

[Article by V. V. Latyshev, V. G. Guryanov, M. Yu. Asliddinova, I. I. Cholovyan, and G. I. Mironova, Donetsk State Medical Institute imeni M. Gorki.]

UDC539.219.3:546.11:539.21

[Abstract] It has previously been demonstrated that hydrogen is capable of stimulating the diffusion of contaminants in metals in those cases where the contaminant atoms are introduced to the metal conjointly with hydrogen. However, the experimental conditions under which this was observed were not under equilibrium. In the present work a study was made of the effects of hydrogen on the mobility of contaminants under relatively mild thermodynamic conditions, viz. saturation of a metal from the gaseous phase. This has practical significance for optimization of preparing ultra-high purity hydrogen (containing less than 10^{-7} to 10^{-8} parts by volume of impurities). Hydrogen having this degree of purity may be obtained by diffusion through non-porous palladium alloy membranes. In the present work a double diffusion filtration cell was used to determine the contamination sources of diffusion purified hydrogen. It was demonstrated that the diffusion rates of contaminants (C, N, and O) could be significantly increased by the simultaneous diffusion of hydrogen. Figures 2; references 4 (Russian).

An Investigation of the Effect of the Conditions of the Preparation of a Porous Surface Layer on the Hydrogen Permeability of Membranes Made of Palladium-Ruthenium Alloys

927M0022B Moscow *ZHURNAL PRIKLADNOY KHIMII* in Russian No 6, Jun 91 (manuscript received 8 May 90) pp 1308-1312

[Article by M.Ye. Sarylova, A.P. Mishchenko, V.M. Gryaznov, and I.A. Litvinov, Petrochemical Synthesis Institute imeni A.V. Topchiyev, USSR Academy of Sciences, Moscow]

UDC 541.183:541.128:534.121.2:546.98-19+546.96

[Abstract] The authors of the study reported herein examined the possibility of creating a porous layer on the surface of membranes made of palladium alloy in order

to clarify the effect of their nature on their hydrogen permeability. The experiments were performed on 1- μm -thick specimens of foil made of alloys of palladium with 6.0 and 9.8% (by mass) Ru or else with 4.8% (by mass) Ru and 0.6% (by mass) La. The foils were prepared as described elsewhere. Porous layers were then applied to the membrane surfaces by thermal diffusion and chemical treatment using Zn, Hg, and Cu. Mercury was applied to the cleaned foil in the form of a liquid, and zinc was applied electrochemically. The copper plating was accomplished from alkaline cyanide electrolytes at 328 K. To achieve mutual diffusion of the substrate material and the applied metal, the authors heated the coated foil in Ar or H₂ under a pressure of 0.1 MPa or at 10⁻²Pa at the specified temperature. The specimens were then cooled to about 20°. The membranes with zinc layers were held from 473 to 723 K, after which the zinc was removed by hydrochloric acid. The copper was extracted by treating the membranes with a CCl₄COOH solution. X-ray photoelectron spectroscopy studies were performed on the specimens. It was discovered that surface treatment with Hg, Zn, and Cu increases the rate of hydrogen transfer by a factor of 1.5 to 2 in the temperature range studied. Increasing the thickness of the metal layer applied to the surface of the Pd-Ru alloy to 4-5 μm was found to increase membranes' hydrogen permeability. Applying a thicker layer of Hg was found to reduce hydrogen permeability. Further studies conducted by using Zn and Cu at a layer thickness of 4-5 μm revealed that the most hydrogen-permeable membranes turned out to be those produced by heat treatment in the temperature range from 500 to 550 K by using zinc and those produced with heat treatment in the temperature range from 560 to 570 by using copper. Figures 5, tables 5; references 6: 5 Russian, 1 Western.

An Investigation of the Effect of Thermal Diffusion and Chemical Treatment of the Surface of Membrane Catalysts Made of Alloys of Palladium With Ruthenium and Rhodium on the Hydrogenation of Pentadiene-1,3

927M0022C Moscow ZHURNAL PRIKLADNOY KHIMII in Russian No 6, Jun 91 (manuscript received 8 May 90) pp 1312-1316

[Article by A.P. Mishchenko, V.M. Gryaznov, and M.Ye. Sarylova, Petrochemical Synthesis Institute imeni A.V. Topchiyev, USSR Academy of Sciences, Moscow]

UDC 541.183:541.128:534.121.2:546.98-19+546.96:542.941.7:547.315.5

[Abstract] The authors of the study reported herein examined the effect of thermal diffusion and chemical treatment of the surface of membrane catalysts. They did so by studying the said catalysts' activity and selectivity in the hydrogenation of pentadiene-1,3. To prepare the membrane catalysts, the authors used specimens of foil 100 μm thick made of alloys of Pd with 5.0% (by mass) Rh (PdRh5) and 15% (by mass) Rh (PdRh15) and with

5.9% (by mass) Ru (PdRu6) and 9.8% (by mass) Ru (PdRu10). Layers of Zn, Cu, Ga, or Hg about 5 μm thick were applied to the foil specimens' surfaces. The membranes with the zinc layer were held for 1 hour at 523 K, and those with a copper layer were held at 673 K in an argon atmosphere. The membranes with a gallium or mercury layer were held for 3 hours at 323 K. The Zn, Ga, Cu, and Hg were then removed by solutions of different reagents. The catalytic tests were performed in a flowthrough double-chamber reactor with a membrane geometric surface of 24 cm². H₂ or a mixture of H₂ and argon was fed through from one side, while pentadiene-1,3 vapors in argon were fed from the other side. The pressure in the reactor on both sides of the membrane equaled 0.1 MPa. The starting pentadiene-1,3 was cleansed of traces of sulfur-containing compounds by passing vapors through a saturated NaOH solution. The products were subjected to infrared chromatography studies. The studies established that it is predominantly pentane, small quantities of pentenes, and up to 3.5 mol% cyclopentene and cyclopentane that are formed on the surface of a catalyst with a porous surface. The main hydrogenation product formed on a catalyst with a smooth surface is pentene-2 (as has been shown elsewhere). An examination of the dependence of pentene selectivity on conversion during the hydrogenation of pentadiene-1,3 revealed that the nature of the metal used to loosen the surface affects the properties of a membrane catalyst. Gallium-treated specimens were found to possess the greatest pentene selectivity. Thermal diffusion and chemical treatment of the surface of membranes made of the alloy PdRh15 with zinc also lead to the appearance of cyclic hydrocarbons in the catalysis product. This phenomenon was not observed in after surface treatment with mercury or copper. Membranes made of a Pd-Rh alloy had a lower pentene selectivity than did those made of the Pd-Ru alloy. Surface treatment with copper did raise the pentene selectivity of the PdRh15 membrane sharply, however. The authors attributed the change in membrane catalyst selectivity occurring after surface treatment with Zn, Ga, or Cu with the formation of new active centers. Figures 4, table 1; references 8: 6 Russian, 2 Western.

Chemically Initiated Electron Transfer Luminescence of Ru(bpy)₃Cl₂ in a Catalytic Reaction With 1,2-Dioxethane

927M0022D Moscow ZHURNAL PRIKLADNOY KHIMII in Russian No 6, Jun 91 (manuscript received 8 Feb 90) pp 1316-1321

[Article by A.I. Voloshin, G.L. Sharipov, V.P. Kazakov, and G.A. Tolstikov, Chemistry Institute, Bashkir Scientific Center, Ural Department, USSR Academy of Sciences, Ufa]

UDC 539.379:541.128:541.49:546.962:542.92:547.518

[Abstract] The authors of the study reported herein conducted a study to prove the presence of chemically

initiated electron transfer luminescence during the breakdown of stable adamanthyl-containing dioxethane in the presence of $\text{Ru}(\text{bpy})_3\text{Cl}_2$. The study compounds were produced in accordance with methods described elsewhere. An FEU-140 multiplier phototube was used to record chemiluminescence. The chemi- and photoluminescence spectra were recorded on a unit equipped with an SSR-1 monochromator and FEU-119 multiplier phototube. The kinetic parameters of the dark decomposition of 1,2-dioxethane in the presence of Ru (II) were measured at temperatures between 113 and 117° by using sealed ampules in steel jackets. At specified time intervals (every 0.75 to 1 hour), the ampules were removed and quickly cooled to a temperature of 75°. The intensity of photoluminescence was measured, and the procedure was repeated. The intensity of photoluminescence was proportional to the concentration of nondecomposed 1,2-dioxethane. The quantum photoluminescence yield of Ru (II) in air-saturated and degassed MeCN was determined based on a quinine standard. The excitation yields of the ketones formed during the decomposition of 1,2-dioxethane were determined by the chemiluminescence method. The error of measuring the yields was less than 25%. The studies performed confirmed that the catalysis and activation of the chemiluminescence of 1,2-dioxethanes by a ruthenium complex occurs in accordance with the mechanism of chemically initiated electron transfer and that this mechanism results in the effective excitation of $\text{Ru}(\text{bpy})_3\text{Cl}_2$ with an efficiency (Φ_{Ru}) of 0.20 ± 0.05 . Figures 3, tables 2; references 12: 4 Russian, 8 Western.

High-Temperature Oxidation of Ti-Fe Alloys in an Air Atmosphere

927M0024A Leningrad ZHURNAL PRIKLADNOY KHMII in Russian Vol 64 No 3, Mar 91 (manuscript received 24 Sep 90) pp 473-480

[Article by V.I. Dyachkov, Leningrad State University]

UDC 620.193.5:669.295.5

[Abstract] A great number of research works have focused on the question of the oxidability of titanium-iron alloys. The insufficiency and discrepancies in the data that do exist have led many researchers to consider the question still open. In view of this fact, the authors of the study reported herein conducted a set of experiments to establish the effect of iron on the nature of the kinetic laws governing the oxidation of titanium and the distribution of oxygen between the metal and oxide phases. The oxidation kinetics of Ti-Fe alloys were studied by the method of precision gravimetry in a static air medium with controlled humidity in the temperature interval from 1,073 to 1,473 K. Alloys containing 1.50, 5.0, 7.0, and 10.0% (by mass) iron were prepared in accordance with the standard technology for titanium. TG-00 titanium sponge and Armco iron were used. Specimens ranging from 2 to 10 mm in thickness that had been cut from 12-mm-diameters cylindrical bars

were subjected to a variety of tests (including metallographic, electronographic, and roentgenographic analysis). The results obtained generally confirmed other published data but did contradict findings published by yet other researchers. The following are among the main conclusions drawn in the present study. The dependence of the heat resistance of Ti-Fe alloys with the aforesaid iron contents and in the aforesaid temperature range is complex and frequently not monotonous. The oxidation kinetics of the alloys studied appears to be subordinate to Evans' equation, which confirms the nonstationarity of the process. The dross on Ti-Fe alloys consists mainly of rutile along with some amount of the FeTiO_3 formed at high temperatures located at the metal interface. The electronographic and roentgenographic analyses performed indicate that the process of oxidation of Ti-Fe alloys at high temperatures results in the iron enrichment of both the surface zone of the metal and the inner and outer layers of the oxide film. The authors were further led to conclude that alloying titanium with iron under the temperature conditions studied causes an increase in the fraction and dissolution rate of the oxygen in the metal phase and in an increase in the role of diffusion in controlling the oxidation rate and the degree of participation of the interstitial titanium ions in the diffusion dross formation process. These changes are in turn attributed to Fe alloying-induced changes in the titanium's heat resistance and in the chemical makeup of the structure of its oxide coating and the gas-saturated zone of the metal. Figures 3, tables 2; references 16: 10 Russian, 6 Western.

X-Ray Spectroscopy of Surface of Chemically Deposited Nickel Coatings

927M0024B Leningrad ZHURNAL PRIKLADNOY KHMII in Russian Vol 64 No 3, Mar 91 (manuscript received 12 Feb 90) pp 519-524

[Article by V.V. Yasulaymene, A.A. Sudavichyus, and Ya.I. Valsyunene, Chemistry and Chemical Technology Institute, Lithuania Academy of Sciences]

UDC 621.357.7:535.33

[Abstract] Chemically deposited nickel coatings are often used in place of gold and silver coatings in the manufacture of electronic equipment. Among other things, such coatings must demonstrate good solderability when the appropriate fluxes are used. In view of this fact, the authors of the study reported herein conducted a detailed examination of the surface state of nickel-phosphorus and nickel-boron coatings that have been chemically deposited by using the appropriated reducing agents. The said coatings were studied before and after heat treatment. Also examined were the distribution of the main components of the Ni-P and Ni-B coatings throughout their depth as the thickness of the coating layer increases and the possibility of mutual diffusion between the Ni-P and Ni-B coatings and the VT-1 titanium base after heat treatment. After coating,

the titanium plates were heat-treated in air and in a helium atmosphere. The specimens were heated at a rate of 12°/min to 350° and held at that temperature for 30 minutes. They were then cooled to room temperature over the course of 6 hours. The specimens were then subjected to x-ray photoelectron spectroscopy studies. It was discovered that heat treating nickel coatings in an air atmosphere results in structural and various behavioral changes in the nonmetals in the surface layer. In the case of Ni-P coatings, the phosphorus on the coating surface was found to oxidize and volatize from a surface layer about 20 nm thick. The Ni-P coatings that had been heat treated in a helium atmosphere turned out to be very different from those treated in an air atmosphere. The x-ray photoelectron spectra of those specimens treated in the helium atmosphere were found to correspond to those of specimens that had not been subjected to heat treatment at all. In the case of Ni-B coatings heat-treated in an air atmosphere, the boron and its compounds were discovered to diffuse to the coating surface and oxidize completely. These differences were deemed responsible for the differing solderability of Ni-P and Ni-B coatings. Figures 3, table 1; references 14: 7 Russian, 7 Western.

Estimation of the Role of Electrolytes' Diffusing Power During Electrochemical Treatment of Long Products With Thicknesses on the Order of Microns

927M0024C Leningrad ZHURNAL PRIKLADNOY KHIMII in Russian Vol 64 No 3, Mar 91 (manuscript received 16 Mar 90) pp 528-531

[Article by T.A. Voronina, S.N. Sirotkin, and A.V. Pomosov]

UDC 621.357.8.035

[Abstract] The existing methods of measuring the diffusing power of electrolytes cannot be used for the working conditions during commercial electrolysis. The diffusing power values thus obtained provide only comparative data for a corresponding estimate of the given electrolyzer or electrolysis mode, and completely specified standard geometric conditions must be specified for such an estimate. The resultant measurements apply solely to the average current density calculated according to two very different values, which must be considered inconsistent. Furthermore, all of the formulas that have been proposed for the quantitative characteristics of diffusing power are tied to one measurement method or another and are all arbitrary to a certain degree. In view of these and other difficulties, the authors of the study reported herein performed a series of experiments designed to clarify the role of the diffusing power of electrolytes during the electrochemical treatment of long products with thicknesses on the order of several microns. They examined electrolytes for electrochemical degreasing and polishing based on orthophosphoric acid, a sulfate-phosphate polishing electrolyte, and several zinc-plating electrolytes. This examination revealed that

several factors have a deleterious effect on electrolytes' diffusing power. Included among these are the following: reducing the thickness of the processed product to several microns, using highly conductive electrolytes, increasing the length of the live zone, and increasing the resistivity of the product's metal. Next, the authors examined the effect that reducing an electrolyte's conduction of electricity would have on its diffusing power. They did so by studying the example of treating Nichrome wire (10 and 20 μm in diameter) in an electrolyte based on 70% H_3PO_4 with a block copolymer additive. The electrolyte's diffusing power did indeed rise. The authors acknowledge, however, that the main goal is not increasing an electrolyte's diffusing power in and of itself but rather to provide quality surface treatment. They further acknowledge that increasing an electrolyte's diffusing power is a necessary but insufficient condition of high-quality surface treatment. These facts lead the authors to recommend that electrolytes with negative diffusing power values must not be used with products having thicknesses of a few microns if these products have a high resistance. Electrolytes with a higher resistivity should be used instead. Figures 4; references 5 (Russian).

Laws Governing Continuous Gas Extraction in a Countercurrent Mode

927M0024E Leningrad ZHURNAL PRIKLADNOY KHIMII in Russian Vol 64 No 3, Mar 91 (manuscript received 28 Jan 90) pp 569-573

[Article by A.N. Marinichev and A.S. Bureyko]

UDC 543.544

[Abstract] Continuous gas extraction is one of the most popular versions of gas chromatographic vapor-phase analysis. Generally speaking, when the method is used, the liquid phase remains in place while the gas flow contacting the liquid extracts the volatile substances being studied. In a countercurrent mode, both the gas and liquid phases move in opposite directions, with the phases configured either vertically or horizontally. This article reports a mathematical analysis of the laws governing the countercurrent mode of continuous gas extraction of matter from a solution. Two methods of establishing the time dependence of the initial mass transfer concentration of volatile matter in a liquid-phase flow moving through a vessel at a given velocity are presented. Also provided are an expression specifying the time dependence of the concentration of matter in the gaseous phase at the vessel's outlet and expressions for using the resultant value as a basis for obtaining other process parameters (such as the gas velocity and gas space over the liquid flow). The expressions presented are checked by means of model calculations of selected process parameters. The starting and reduced parameter values in four of the nine computation results presented are very different from one another. In the other cases presented, the calculated values reproduced the starting

values with a rather high precision (about 1%). Tables 2; references 9: 6 Russian, 3 Western.

An Investigation of the Glass Range in the System PbO-B₂O₃-ZnO To Manufacture Vacuum Tube Connectors

927M0024J Leningrad ZHURNAL PRIKLADNOY KHIMII in Russian Vol 64 No 3, Mar 91 (manuscript received 29 May 90) pp 656-658

[Article by N.V. Konicheva, L.V. Ivanova, P.I. Buler, and I.D. Kashcheyev, Ural Polytechnic Institute imeni S.M. Kirov]

UDC 666.1.037.546.621

[Abstract] Glasses of the system PbO-B₂O₃-ZnO may be used in manufacturing vacuum tube connectors to serve as an insulator sealing the connecting aluminum casing with the lead. One problem with using the said glasses to manufacture electric connectors is that they tend to crystallize during heat treatment. This in turn results in the instability of such parameters characterizing the glasses as their thermal coefficient of linear expansion, viscosity, conduction, and water resistance. For this reason, the authors of this concise report conducted a study to determine the range of the existence of glasses in the system PbO-B₂O₃-ZnO that will crystallize under the temperature conditions of the manufacture of electric connectors. The study glasses were synthesized in a platinum crucible from chemically pure and graded-for-analysis materials in an electric muffle furnace at 900 to 950° for 30 to 45 minutes. A total of 40 different glass compositions were synthesized. Each specimen (mass, 0.2 g) was subjected to crystallization at 450-470° for 30 minutes. Five specimens were determined to have no signs of crystallization, and four were found to have only minimal signs of surface crystallization. These were singled out for further study. The curves of the concentration dependences of the properties of the study glasses reveal that both resistivity and water resistance reach their maxima at a concentration of 50 mol% PbO. A sharp drop in viscosity also occurs when the PbO content is increased from 40 to 50 mol%. Above 50 mol% PbO, viscosity continues to drop but does so less intensively. A change in the coordination state of boron occurs simultaneously with the change in the structural role of lead in the said glasses. The fraction of B₄ increases to 0.5 at 50 mol% PbO and then drops sharply (because the lead cation ceases to be an oxygen donor). The thermal coefficient of linear expansion drops as the PbO content increases from 50 to 60 mol%. At 20°, the glasses' resistivity is between 7.6 and 17 x 10¹² ohms/m and is also extremely dependent on the PbO content. Increasing the PbO content to 50 mol% also increases resistivity. Finally, the water resistance of the glasses studied is relatively low (between 95 and 96%) and reaches an obvious maximum at 50 mol% PbO. Figures 3; references 5 (Russian).

The Effect of Thallium on the Superconductivity Properties of High-Temperature Superconductors

927M0025A Moscow ZHURNAL NEORGANICHESKOY KHIMII in Russian Vol 36 No 6, Jun 91 (manuscript received 14 Sep 90) pp 1363-1367

[Article by N.S. Kopelev, M.Ye. Zalishchanskiy, and Yu.M. Kiselev, Moscow State University imeni M.V. Lomonosov]

UDC 541.123.31

[Abstract] The authors of the study reported herein examined the effect of indium and gallium oxides on the superconductivity properties of high-temperature superconductive thallium ceramics. Specifically they study the effect of indium and gallium oxides in two composites: Tl_{2.0-x}M_xBa₂Ca₂Cu₄O_y and Tl_{1.5-z}M_zBa₂Ca₃Cu₄O_y (where M = In, In + Ga, and Ga; x = 0.0 to 1.3; z = 0.0 to 1.0; and Δx = Δz = 0.1). As starting materials the authors used high-purity thallium, indium, and gallium oxides; graded-for-analysis barium carbonate; chemically pure barium and calcium nitrides; and graded-for-analysis copper, barium, calcium, and indium nitrates. Calcium oxide was obtained by thermal decomposition of graded-for-analysis CaCO₃ at 850°C in air for 5 hours. The compounds synthesized were studied by the methods of magnetochemistry and chemical analysis. The studies performed revealed that the critical temperature of the superconductivity transition of the study compounds decreases as the thallium content increases. The increase was most evident in the case of the thallium-gallium systems studied (critical temperatures of 83 and 82.5 K, respectively). Preparations synthesized in the system Tl-In-Ba-Ca-Cu-O with an analogous makeup were found to be characterized by higher critical temperatures (98.5 and 94 K, respectively). The thallium-indium specimens studied were also found to preserve their superconducting properties with a lower thallium content (In:Tl = 1.5) than in the case of their thallium-gallium analogues (Ga:Tl = 1.0). The critical temperatures of the Tl_{2.0-x}M_xBa₂Ca₂Cu₄O_y specimens studied were found to be about 5 to 10 K higher than those of the respective specimens of the series Tl_{1.5-z}M_zBa₂Ca₃Cu₄O_y. Specimens with a low thallium content were found to be characterized by weak Josephson contacts. Heat treatment (raising the synthesis temperature from 870°C to 910°C) was found to increase the critical temperature of all of the specimens studied. Heat treatment in the temperature range from 910 to 930°C did not appear to cause any significant changes in critical temperature. Further temperature increases cause the disappearance of superconducting properties in the study specimens, however. Repeated heat treatment was found to lower the critical temperature and, in the case of specimens with a thallium content less than 65 mol%, to result in the disappearance of superconducting properties. The prehistory of the thallium-free charge was also found to have a significant effect on critical temperature. The best results were achieved in the case of a

charge prepared by the ceramic method from the respective oxides and BaCuO_2 . Figures 3, tables 4; references 5: 1 Russian, 4 Western.

The Production and Properties of the Phase 2223 in the System Bi-Pb-Sr-Ca-Cu-O

927M0025B Moscow ZHURNAL
NEORGANICHESKOY KHIMII in Russian
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pp 1368-1371

[Article by N.M. Drozdova, I.S. Dubenko, A.A. Yevdokimov, A.G. Laptev, S.A. Smirnov, and Yu.V. Titov, Moscow Radio Engineering, Electronics, and Automation Institute]

UDC 291.19.29.10.12.46

[Abstract] The authors of the study reported herein worked to produce single-phase specimens with the structure 2223 in the system Bi-Pb-Sr-Ca-Cu-O . They then proceeded to study the said phase's makeup and superconductivity properties. The synthesis was performed via by means of the conventional ceramic technology in air. High-purity SrCO_3 , CaCO_3 , Bi_2O_3 , CuO , and PbO were used as starting materials. After homogenization, all of the specimens were annealed at 750°C for 10 hours, wiped repeatedly, heat treated for 50 hours at 800°C, homogenized once more, pressed into tablets, and held at 845-855°C for several days. The specimens were subjected to x-ray phase and chemical analyses, and their electrophysical and magnetic properties were measured. The studies performed revealed that the phase 2223 makes the transition to a state of superconductivity at 116 K. The studies also confirmed that the true makeup of phase 2223 differs from the ideal state. For that reason, to obtain 2223 monocrystals by the recrystallization method the authors used a starting composition of $\text{Bi}_{1.68}\text{Pb}_{0.32}\text{Sr}_{1.8}\text{Ca}_{1.8}\text{Cu}_{2.8}\text{O}_y$ and performed the annealing at 855°C for 720 hours. The resultant tablet consisted entirely of monocrystals up to 60 μm in diameter, with the phase 2223 constituting 95% and with a temperature of the transition to superconductivity of 105 K. Metal distribution studies based on 29 phase 2223 monocrystals revealed that lead replaces the bismuth atoms in doubled layers and even Bi, Sr, and Ca atoms simultaneously. The strontium position was found to be simultaneously the most stable and the closest to the ideal composition. The Bi, Ca, and Cu positions were less mobile. Further research is required, however, to definitively determine which are replaced by lead to the greatest degree. Figures 3; references 20: 1 Russian, 19 Western.

Dielectric Properties in the System $(1 - x)\text{BaTiO}_3\text{-}x(\text{Ba}_{2/3}\text{Y}_{1/3})\text{CuO}_{3.8}$ ($0 \leq x \leq 0.5$)

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[Article by G.M. Kaleva, M.V. Kudinova, S.G. Prutchenko, Ye.D. Politova, S.Yu. Stefanovich, and Yu.N. Venevtsev]

UDC 537.226.4:666.3

[Abstract] The authors of the study reported herein examined the dielectric properties of compounds in the system $(1 - x)\text{BaTiO}_3\text{-}x(\text{Ba}_{2/3}\text{Y}_{1/3})\text{CuO}_{3.8}$. Because they studied the said system from the standpoint of barium titanate ($x = 0$ to $x = 0.5$), they used the conventional methods of analyzing the ferroelectric state, i.e., dielectric measurements at different frequencies and the method of generating the second harmonic of laser radiation. The study specimens were produced by solid-phase synthesis. Silver electrodes were applied to the specimens to permit the taking of dielectric measurements. The ceramic specimens' microstructure and makeup were monitored by using a JEOL-35CF electron microscope and x-ray microspectrum analyzer. The studies performed on the specimens of a solid solution ($0 \leq x \leq 0.02$) of the study ferroelectric revealed that the solution's ferroelectric properties attenuate in a regular fashion as x increases. This finding was interpreted as confirmation of the opposite effect of crystalline chemical factors (makeup, chemical bond) on the conditions of the realization of the ferroelectric state on the one hand and superconductivity on the other hand. The concentration dependence of the intensity of the second harmonic in the range of high x did not turn out to drop as sharply as might be expected. Rather, it demonstrated a tendency toward stabilization or even somewhat of an increase. This behavior was interpreted as an indication of the high polarizability (including nonlinear polarizability) of the main components of the heterophase mixture that appears in the presence of the effect of generation of the second harmonic for pure $\text{YBa}_2\text{Cu}_3\text{O}_7$, i.e., for a composition of $x = 1$ of the system under consideration. Figures 4; references 14: 5 Russian, 9 Western.

An Investigation of the Structure and Electrophysical Properties of Quick-Hardened Cadmium Antimonide Foils

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[Article by V.G. Shepelevich, Belorussia State University imeni V.I. Lenin, Minsk]

UDC 548.5:621.315

[Abstract] Cadmium antimonide (CdSb) semiconductor compounds are widely used in instrument making, and the structure and electrophysical properties of CdSb poly- and monocrystals, layers, and films produced by conventional methods have been researched. The authors of the study reported herein examined the structure and electrophysical properties of CdSb foil obtained by quick-hardening from the liquid state. The CdSb foils studied were obtained by crystallizing a drop of melt (about 0.3 g) located on the inside polished surface of a rotating copper cylinder. The foil produced

had a thickness between 20 and 60 μm . It was cooled at a rate of no less than 10^6 K/s . The foil specimens were subjected to metallographic, x-ray microanalysis, and x-ray crystallographic studies. A monotonic increase in both the foil's resistivity (ρ) and differential thermoelectromotive force (α) was observed to occur as the temperature was increased. The Hall coefficient (R) amounted to $1.3 \times 10^{-9} \text{ m}^3/\text{C}$. The positive signs of α and R indicate that the transfer processes are determined by the holes, the concentration and Hall mobility of which equal $5 \times 10^{27} \text{ m}^{-3}$ and $1.5 \times 10^{-4} \text{ m}^2(\text{V}\cdot\text{s})$, respectively, at 77 K. The large concentration of holes is explained by the temperature course of the resistivity, as are the low values of ρ , R , and α . The authors hypothesize that antistructural defects are responsible for the high concentration of holes. The quick-hardened CdSb foils produced were found to be unstable and to exhibit changes in their electrical properties during heating. Isothermic annealing of the quick-hardened foil at temperatures of 393-473 K was found to cause a monotonic increase in α . Annealing also caused an increase in ρ and R . When the CdSb foil was heated above 380 K, the cadmium atoms began migrating throughout the crystal, which in turn resulted in the disappearance of antistructural defects. A change in α was observed when the CdSb foil was heated above 610 K; this change was associated with the vaporization of cadmium, which in turn resulted in the destruction of the specimens. The study results led the authors to conclude that from the standpoint of their properties, CdSb foils produced by quick-hardening from a melt occupy an intermediate position between monocrystals and films produced by thermal sputtering and may be used in instrument making. Figures 2; references 5: 4 Russian, 1 Western.

The Mechanism of the Dissolution of Copper (II) Oxides and $\text{YBa}_2\text{Cu}_3\text{O}_{7-\text{x}}$ in Acidic Media

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[Article by D.A. Zenchenko, I.G. Gorichev, I.S. Shaplygin, and V.D. Serokhov, General and Inorganic Chemistry Institute imeni N.S. Kurnakov, USSR Academy of Sciences]

UDC 532.73.45:49

[Abstract] The mechanism and kinetics of the dissolution of metal oxides in various media have been discussed numerous times. Few data are available on the effect that pH, complexing agents, anions, and other factors have on the mechanism of the dissolution copper (II) oxide and $\text{YBa}_2\text{Cu}_3\text{O}_{7-\text{x}}$. In view of this fact, the authors of the study reported herein studied and discussed the mechanism of the dissolution of the said compounds from the standpoint of an acid-base model of the structure of a binary electric layer at the oxide-electrolyte interface. They placed weighted portions of

high-purity copper oxide and $\text{YBa}_2\text{Cu}_3\text{O}_{7-\text{x}}$ in a thermostatted reaction vessel containing 1 liter of an aqueous acid solution (HCl , H_2SO_4 , HClO_4) at different pH levels. To eliminate diffusion problems, the experiments were performed in a mode of free feed of the oxide particles (about 500 rpm) at different temperatures and sulfuric and perchloric acid concentrations. The studies performed indicated that the maximum rate of dissolution of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\text{x}}$ is 1.5 to 2 orders of magnitude higher than that of copper (II) oxide. The equilibrium constant characterizing proton bonding was also found to be higher in the case of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\text{x}}$. The said constant was determined to depend on the pH of the solution, and the ions' transition from solid phase to solution was found to depend on potential. The acid-base model proposed enabled the authors to explain all of the kinetic features of the dissolution of copper oxide phases. The differences between the study compounds with respect to the orders of magnitude of their hydrogen ions were attributed to the fact that the dissolution rate of the oxide phase should indeed be expended to increase as the positive potential increases. The authors were also able to clarify the activating role of anions in the process of the dissolution of copper (II) oxide: They attributed the increase in the dissolution rate of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\text{x}}$ versus that of copper (II) oxide to a shift in pH to the acid range and a reduction in the coefficient of proton bonding. Giving consideration to the electrostatic jump in potential occurring and the oxide-electrolyte interface will not result in any significant change in the kinetic equations and parameters of the dissolution process. Figures 4, tables 2; references 20: 12 Russian, 8 Western.

Synthesis and Structure of Strontium and Calcium Mercurates

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[Article by S.N. Putillin, M.G. Rozova, D.A. Kashporov, Ye.V. Antipov, and L.M. Kovba]

UDC 546.492.41.42

[Abstract] From a formal standpoint, oxygen compounds of mercury are subdivided into compounds of univalent mercury that generally contain a metal-metal bond and compounds containing bivalent mercury proper. Only very complex oxides of bivalent mercury are known (for example, mercurates of alkaline metals of the type A_2HgO_2 , where $\text{A} = \text{Li-Cs}$). The authors of the study reported herein examined the possibility of synthesizing complex oxides of mercury with other elements, specifically with oxides of alkaline earth metals. For their studies they selected SrO and CaO produced by roasting graded-for-analysis strontium and calcium carbonates at 800° for 24 hours in a vacuum along with graded-for-analysis HgO . Specimens were prepared with MO:HgO ratios of 1:1,

1:1.5, and 1:2. The synthesis was performed at a temperature of 550°C over the course of 24 hours. The resultant specimens were subjected to x-ray analysis. The specimens with 1:1.5 and 1:2 ratios of MO and HgO did not turn out to be single-phase compounds; their roentgenograms contain lines belonging to HgO. The SrHgO₂ produced from a 1:1 ratio of MO and HgO were yellow. Its elementary cell proved to have a rhombohedral limiting boundary and parameters of $a = 3.8273(7)$ and $c = 18.997(4)$ angstroms. The CaHgO₂ produced had parameters of $a = 3.583(1)$ and $c = 18.641(7)$ angstroms. X-ray crystallographic studies performed on the compounds synthesized revealed that their stoichiometry and parameters are similar to those of KRO₂ (R = La-Yb) complex oxides. Unlike these R atoms, however, the Hg atoms in the study compounds have a coordination circle in the form of a dumbbell. This coordination pattern is typical for bivalent mercury in oxide compounds having strongly covalent bonds with axial oxygen atoms. In the structure studied they were determined to form on account of a shift in the layer of oxygen atoms perpendicular to the c axis. This fact was interpreted to mean that the mercury atoms unite pairs of oxygen atoms from two densely packed anionic layers and was deemed similar to the dumbbell-like circle for Hg²⁺ realized in M₂HgO₂ structures (M being an alkaline metal) derived from the structure K₂NiF₄. Figures 2, tables 2; references 4 (Russian).

Phase Diagrams and Electrophysical Properties of Alloys of the System Bi₂X₃-ZnX (X = S, Se, Te)

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[Article by I.N. Odin and V.V. Marugin, Moscow State University imeni M.V. Lomonosov]

UDC 546.221'23'24'47'87+537.32

[Abstract] The authors of the study reported herein have previously published phase diagrams of the systems Bi₂Se₃-ZnSe and Bi₂Te₃-ZnTe. Several segments of those diagrams were not investigated at the time, however. In the present article, the authors investigate those phase diagram segments that they did not study before. Specifically, they examine the behavior of the said compounds at temperatures above 1,450 K and in the concentration ranges of 50-99.5 mol% ZnSe or 77-99.5 mol% ZnTe. In addition, they study the system ZnS-Bi₂S₃ and determine the electrophysical properties of alloys of the system Bi₂X₃-ZnX (X = S, Se, Te). The study alloys were synthesized as described previously from stoichiometric quantities of Bi (type V0000) and sulfur (high-purity 16-5). Monocrystalline ZnS with a total impurity content of 10⁻³% (by mass) was used. The specimens were subjected to differential thermal analysis, x-ray phase analysis, and microstructural analysis. The following are among the main findings of the analyses performed. A solid solution γ forms on the basis

of Bi₂S₃, and the lines of the solidus and liquidus pass through a maximum at the coordinates T = 1,065 K, 3.5 +/- 0.5 mol%, ZnS. At a concentration of more than 4 mol%, the lines of the liquidus and solidus drop to the eutectic temperature of 1,047 +/- 6 K. The ZnS content at the eutectic point is 6 +/- 1 mol%. The system Bi₂Se₃-ZnSe has a eutectic-type phase diagram (the coordinates of the eutectic point are 973 +/- 5 K and 6 +/- 1 mol% ZnSe), whereas the system Bi₂Te₃-ZnTe has a peritectic-type phase diagram (the coordinates of the peritectic point are 4 +/- 1 mol% ZnTe, 871 +/- 4 K). Adding 0.7 mol% ZnTe to an alloy of the system Bi₂Te₃-ZnTe changes the conduction from hole (for Bi₂Te₃) to electron conduction. Zinc selenide is also a donor additive to Bi₂Se₃. Figures 4; references 4 (Russian).

The Phase Complex of KCl + NaVO₃ in a Reversible Reaction With NaCl + KVO₃

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pp 1847-1853

[Article by A.S. Trunin, A.S. Petrov, O.V. Temirbulatova, and I.K. Garkushin, Kuybyshev Polytechnic Institute imeni V.V. Kuybyshev]

UDC 541.123.5:543.226

[Abstract] The authors of the study reported herein used a complex methodology to study the phase complex of KCl + NaVO₃ in a reversible reaction with NaCl + KVO₃. The said methodology was designed to enable the authors to clarify the phase complex of the said system and the characteristics of its non-, mono-, and divariant equilibrium so as to develop a quantitative description of these equilibria. The following analysis methods were used: differential thermal analysis [DTA], complex DTA (combined writing of the DTA and electric conduction curves), and x-ray phase analysis. An F-116/1 microvolt-microammeter photoamplifier was used to amplify the thermoelectromotive force of the differential thermocouple, an IRN-64 controlled voltage source was used to shift the lines of the differential curve, and a DRON-2.0 diffractometer was used to take the roentgenograms. All of the salts used were graded for analysis. The aforesaid reversible reaction was studied on three information levels, i.e., the zero, first, and second levels. The zero-information level refers to the input information, i.e., the classification and characteristics of the starting sodium and potassium chlorides and metavanadates. The first information level refers to a qualitative description of the reaction of the starting substances, and the second information level refers to a quantitative description of the same reactions. In their analysis at what they term the second information level, the authors calculate the characteristic of the nonvariant points of the system and determine the characteristics of its mono- and divariant equilibria. They present a set of three nonvariant and monovariant reaction equations for the three-component

mutual system studied and present seven equilibrium relationships corresponding to the monovariant lines. The data summarized in seven tables are used to plot isotherms of the reactions by using a program written in the language PL/I for the YeS operating system. (Among the items covered in the tables are the elements of the faceting of the study system, data from x-ray phase analysis of the conversion point, possible associations of the system's phases and their stability, formation of possible combinations of phases in the right parts of the exchange-interaction reaction equations, and formation of the left parts of the said equations.) Phase and crystallization trees of the system are plotted. The calculated and experimental data obtained were found to be in good agreement with the maximum error not exceeding 10%. Figures 3, tables 8; references 16 (Russian).

A Gas Chromatographic Study of the Adsorption Properties of Activated Carbon Fibers

927M0031A Moscow *VESTNIK MOSKOVSKOGO UNIVERSITETA: KHIMIYA* in Russian Vol 32 No 3. May-Jun 91 (manuscript received 28 Aug 90) pp 230-234

[Article by G.I. Yemelyanov, L.Ye. Gorlenko, O.A. Malykh, and N.V. Kovaleva, Physical Chemistry Department, Moscow University]

UDC 620.10

[Abstract] Research conducted during the past few years has demonstrated that carbon fiber materials may be used with great success as adsorbents for an entire series of organic compounds. Modifying the surface of activated carbons makes it possible to regulate their adsorption properties over a wide range and to produce adsorbents that are equal or even superior to activated charcoals. In a continuation of this line of research, the authors of the study reported herein used the method of gas chromatography to study the adsorption properties of activated carbons based on cellulose hydrate. Their primary objective in so doing was to find adsorbents capable of fast complete adsorption of toxic organic compounds contaminating the air of industrial sites. As adsorbents, the researchers used two types of carbon fibers (ShL and UL). Each had a large specific surface (about $1,000 \text{ m}^2/\text{g}$) and highly developed microporous structure ($d_{\text{pore}} = 1.5 \text{ nm}$). A Khrom-5 chromatograph with a flame-ionization detector and glass columns 0.5 m long and 0.3 cm in diameter was used in the studies performed. The ShL fiber material placed in the first column weighed 1.0627 g; the UL fiber material placed in the second column weighed 1.299 g. The helium carrier gas was fed through at a rate of 40 ml/min. n-Hexane, benzene, and toluene were used as adsorbates during the adsorption measurements. The amount of adsorbate added was varied from 1 to 10 μl , and the chromatograms were taken in the temperature interval from 280 to 320. The chromatograms obtained indicated

that diffusion and kinetic processes do not play a noticeable role in the adsorption process. In other words, adsorption equilibrium takes place. The isotherms obtained for all of the adsorbates studied were similar in nature. With respect to retained volumes, the adsorbates studied may be ranked as follows for both adsorbents: toluene > n-hexane > benzene, with the retained volumes (v_m , i.e., Henry's constant) at 20° higher for the UL fibers than for the ShL fibers. The dependence of the adsorption heats of n-hexane, benzene, and toluene on the degree of amount of filling was determined for both adsorbents. The shape of the said curves confirms the surface inhomogeneity of the ShL and UL activated carbon fibers. The high initial heat adsorption values for n-hexane and benzene (about twice the values for non-porous graphitized carbon black) are evidently associated with the filling of the finer micropores (whose diameter is commensurate with that of the adsorbed molecules). In the case of toluene, the initial adsorption heats are more than twice those obtained on graphitized carbon black. This may possibly be connected with the additional specific reaction of toluene with the functional groups of the surface of the carbon fiber materials. The adsorption of n-hexane, benzene, and toluene at room temperatures is virtually irreversible. Heat treatment of the fibers at temperatures above 280° is therefore required. Figures 3, table 1; references 6: 3 Russian, 3 Western.

The Electric Conduction Properties of the Fiber VION AN-3 Modified by Transition Metal Ions

927M0031D Moscow *VESTNIK MOSKOVSKOGO UNIVERSITETA: KHIMIYA* in Russian Vol 32 No 3. May-Jun 91 pp 284-288

[Article by A.N. Astanina, N.I. Pleskach, L.N. Serdyukova, M.P. Zhilenko, N.V. Kiryanova, E.M. Trukhan, and A.P. Rudenko, Petrochemistry and Organic Catalysis Department, Moscow University]

UDC 677.4:677-1-17

[Abstract] The authors of the study reported herein worked to develop methods of producing fibers with electronic conduction by including transition metal ions into the polymer matrix. They were especially interested in the possibility of including metal ions by coordination bonding with the polymer's functional groups, which could in turn lead to the formation of complex metal ions in the polymer matrix. Specifically, they studied the electron conduction properties of the polymer fiber VION AN-3 modified by the following transition metal ions: Cu^{2+} , Cr^{3+} , Ni^{2+} , Mn^{2+} , Co^{2+} , Fe^{3+} , and Cd^{2+} . Sorption of the said ions onto the fiber was accomplished from aqueous solutions of nitrates, chlorides, and sulfates of the respective metals. A weighted portion of fiber (0.1 g) was immersed in a 200-ml salt solution with a concentration of 5×10^{-4} and $1 \times 10^{-1} \text{ M}$ and held at room

temperature for 2 hours. The specimens were then filtered out, rinsed with distilled water, and air-dried. The quantity of ions sorbed was determined on the basis of the residual concentration in the sorption solution. It was established that at a sorption solution concentration of 5×10^{-4} M, virtually all of the metal ions are sorbed by the fiber. Only a portion of them are sorbed at a concentration of 1×10^{-1} M. For copper-containing specimens in the former case, the metal ion content amounts to 0.5 mgEq/g. In the second case, it amounts to 1.1 mgEq/g. The difference in the contents of the other ions in the fiber phase upon sorption from diluted and concentrated salt solutions is on the same order of

magnitude (a factor of 3 to 8) as in the case of the copper-containing fibers. The authors demonstrate that the electron conduction properties of transition metal ion-modified VION AN-3 fiber depend on the nature of the metal ion used as a complexing agent, the nature of the sorbed salt anion, the sorption conditions, and subsequent treatment of the metal-polymer fiber by sodium sulfide. The transition metal complexes formed in the polymer phase are demonstrated to possess "island" microwave conduction and may participate in electron transfer processes similar to electron transport chains in biological systems. Figures 2, table 1; references 8 (Russian).

The Liquid-Phase Polymerization of Fluorine-Containing Esters of Acrylates and Methacrylates in a γ -Radiation Field

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[Article by I.P. Kim, I.M. Barkalov, F.A. Baybikov, S.R. Allayarov, and M.G. Rostomkhanov, Chemical Physics Institute imeni N.N. Semenov, USSR Academy of Sciences, Chernogolovka, and Perm affiliate, State Institute of Applied Chemistry]

UDC 541.15:541.127:541.64:547.391.1'26'161

[Abstract] The authors of the study reported herein compared the main kinetic characteristics of the process of radiation liquid-phase polymerization of fluorine-containing methacrylates and their hydrocarbon analogues. The study monomers were purified by distillation to a purity of at least 99.7%. Specimens with five different formulas (detailed in the article) were subjected to calorimetric studies as described elsewhere. Polymerization of the study monomers was initiated by using a ^{60}Co isotope unit. The studies performed revealed that the polymerization kinetics of fluorine-containing methacrylates are well described by a kinetic schematic with a quadratic termination of the growing macromolecules and a distinctly expressed gel effect. It was concluded that adding perfluorinated substituents to the methacrylate molecule likely leads to a reduction of the constant of the rate of breaking of the polymer chains. The kinetics of the polymerization of fluorine-containing acrylates (just like that of their hydrocarbon analogues) was determined to be very different from the polymerization of methacrylates, the main difference being the absence of a quasi-steady state. In other words, the rate of the process was found to increase

sharply from the very outset. This was attributed to the diffusion nature of the termination. Figures 3, table 1; references 5 (Russian).

The Proton Affinity of Phosphoryl Compounds and the σ^{Φ} Constants of Their Substituents

927M0022F Moscow ZHURNAL PRIKLADNOY KHIMII in Russian No 6, Jun 91 (manuscript received) pp 1334-1338

[Article by L.N. Alekseyko, V.V. Gorchakov, M.I. Kabachnik, Ye.I. Matrosov, and S.N. Slabzhennikov, Elementoorganic Compounds Institute imeni A.N. Nesmeyanov, USSR Academy of Sciences, Moscow]

UDC 530.145:941.51:539.125.4:541.63:547.558.1-31

[Abstract] The basicity of phosphoryl compounds in the liquid phase has been the subject of a number of well-founded studies. The basicity of the said compounds in the gaseous phase, i.e., the proton affinity of phosphoryl compounds, has not been studied adequately, however. In view of this fact, the authors of this article undertook such a study. By using the familiar Hammett-type equation $pK_a = pK_a^0 - \rho \sum_i \sigma_i^{\Phi}$, calculations made by the method of partial neglect of the differential overlap, and existing literature data, they were able to demonstrate that the proton affinity of XYZPO phosphoryl compounds is a linear function of the sum of the constants σ^{Φ} . They were also able to determine the center of protonation in the gaseous phase and the nature of the effect of solvents on basicity. Specifically, they conclude that the solvation effect that reduces the basicity of the oxygen atom of a phosphoryl group may be tied to the formation of hydrogen bonds between the atoms of the ester oxygens and the solvent molecules. Figures 3, table 1; references 23: 11 Russian, 12 Western.

Contemporary Processes of Evolving Hydrogen From Oil Refining Gases

927M0015A Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 6, Jun 91 pp 2-3

[Article by Yu.M. Baranov, L.V. Stolyarova, M.F. Shopshin, T.Kh. Melik-Akhnazarov, and T.S. Aleksashkina, Petroleum Refining All-Union Scientific Research Institute]

UDC 661.961.5+62-278

[Abstract] Virtually all fractions of high-sulfur petroleum (with the exception of some amount of tars used to produce bitumens) should in the future be subjected to catalytic refining involving hydrogen. Ninety percent of all oil refined is diverted to hydrofining, hydrocracking, and hydrodesulfurization units. The need for hydrogen has grown accordingly. The conventional source of concentrated hydrogen at oil refineries, i.e., the catalytic reforming process, cannot produce enough hydrogen to desulfurize petroleum products. Current plans are to increase hydrogen production to 1 million tons yearly by the year 2000. About 50% of all the hydrogen-containing gas produced by catalytic reforming (which contains 60 to 80% H₂ by volume) is not of a quality suitable for use. It may, however, be a source of high-purity hydrogen if passed through a hydrogen concentration unit. The conventional cryogenic and adsorption methods of concentrating hydrogen are still the main methods used. Membrane technology is being used increasingly frequently, however. The main drawbacks of the polymer membranes now used are low selectivity with respect to a mixture of H₂, CH₄, and CO₂ and the inability of the membranes to function in the presence of a mix of heavy hydrocarbons. Membranes that are more resistant to these hydrocarbons have been developed and used abroad, however. Besides being ecologically purer than other conventional hydrogen concentration technologies, membrane technology does not require absorbents or reagents and entails lower energy costs. Palladium membranes appear to be a promising alternative to polymer membranes in that they have a higher mechanical strength, are more resistant to corrosion, and have a higher catalytic activity in many chemical reactions involving hydrogen. Series-produced membranes made of palladium alloys have capacities ranging from 100 to 5,000 m³ H₂/h under normal operating conditions. Their main drawback at present is their relatively high cost (compared with polymer membranes), which ranges from 600,000 to 800,000 rubles and which amounts to 70-80% of the cost of implementing palladium membrane-based hydrogen concentration processes. Studies have shown that a palladium membrane-based hydrogen concentration process would be possible with an active layer about 10 µm thick. This would make the technology competitive with other promising hydrogen concentration methods in about 20 to 30 years. Figure 1, tables 2; references 5 (Russian).

The Effect of Catalytic Cracking Conditions on Gasoline Fraction Characteristics

927M0015B Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 6, Jun 91 pp 3-6

[Article by V.A. Stankevich, N.M. Marchenko, T.Kh. Melik-Akhnazarov, N.I. Cherkasova, L.Ya. Vlasenko, L.N. Shabalina, and I.B. Yasnova, Petroleum Refining All-Union Scientific Research Institute and Moscow Oil Refinery]

UDC 665.644.2

[Abstract] The gasoline fraction with an onset of boiling at 195°C is the main end product of the G-43-107 combined catalytic cracking unit of the Moscow Oil Refinery. It is produced from hydrofined vacuum distillate and used as the high-octane component of commercial automotive gasoline. Depending on the cracking conditions used, the yield of the said fraction may reach 46 to 50% (by mass) of the starting material. Its octane number is comparatively low, amounting to 89-89.5 per cracking run (the research octane number) or 80.5-81.5 (the motor octane number). The crude oil used to produce the said gasoline fraction is typically a mixture of Western Siberian and Ukhta (up to 40%) oils. The authors of the study reported herein examined the effect of catalytic cracking conditions on the makeup and octane number of the said gasoline fraction. Tests performed on this mixture in a pilot installation at the refinery revealed that hydrofining the crude oil to a sulfur content of 0.25% (by mass) lowered the gasoline fraction's octane number to 88.5 (the research octane number) or 79.5 (the motor octane number). Reducing the degree of hydrofining from 60 to 10-20% increased the content of olefin hydrocarbons, reduced the content of naphthenic hydrocarbons, and increased the octane number by 1 or 2 points. Next, the researchers attempted to intensify refining of the oil by catalytic cracking of the heavy vacuum distillate. Increasing the temperature of the end of the boiling of the high-sulfur raw material to 550°C reduced the yield of gasoline fraction but increased its octane number by 0.5 to 1.5 points. The effect of the intensity of cracking on the G-43-107 unit was found not to be as significant on the octane number of the gasoline fraction as was the case in the studies performed on the pilot unit. This was evidently due to the fact that the starting material contained a significant amount material that is difficult to convert below 350°C and to the fact that the ratio of Ukhta and Western Siberian crude oils used throughout a cracking cycle varies and thus masks the effect of other factors influencing octane number. Data collected on gasoline fractions produced on the G-43-107 unit revealed that lowering the temperature of the end of boiling from 190 to 140°C only reduced the octane number by 1 to 2 points, depending on the octane number and hydrocarbon makeup of the initial fraction. This decrease was attributed to a reduction in the content of aromatic hydrocarbons that was partially compensated for by an increase in the content of olefin hydrocarbons. An examination of the distribution of sulfur content in the gasoline fraction revealed that beginning at a boiloff temperature of

90°C, the sulfur content in narrow gasoline fractions increases in direct proportion to the boiloff temperature. When the temperature of the end of boiling of the gasoline fraction is reduced from 190 to 140°C, the sulfur content in the fraction decreases by 40%. The data collected on actual gasoline fractions produced in the G-43-107 also confirmed the fact that the content of sulfur in the fractions was indeed reduced by hydrofining of the crude oil. Figures 4, tables 5; references 4 (Russian).

Continuous-Type Operating Model Catalytic Cracking Unit

927M0015C Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 6, Jun 91 pp 6-8

[Article by M.I. Rustamov, G.T. Farkhadova, S.A. Dorofeyev, and N.S. Mayorova, Petrochemical Processing Institute imeni Yu.G. Mamedaliyev, Azerbaijan Academy of Sciences]

UDC 665.644.2.66.096.097.1

[Abstract] The development of new contact-catalytic heterogeneous processes and catalysts generally entails laboratory research and tests on pilot and pilot commercial units. In the case of processes entailing slow catalyst deactivation it is possible to conduct comprehensive studies of key process parameters entirely under laboratory conditions using laboratory equipment. In a number of industrial processes, however, catalysts become deactivated in short periods of time and thus cannot be adequately studied under laboratory conditions. The catalytic cracking of crude oil is one such process. In an effort to reduce the additional time and expense that is generally required to research new catalysts that do not lend themselves to study under laboratory conditions, the authors of the study reported herein developed a new continuous-type model catalytic cracking unit called the Voskhod-4-01. It includes material preparation, reactor-regenerator, reaction production condensation, control, and analytical blocks. Starting material is input at five points along the reactor's height with a semi-throughflow of catalyst. This permits wide variation of the time for which the starting material is in contact with the catalyst and permits the most complete research of the potential activation of newly developed catalysts for contact-catalytic heterogeneous processes. The ability to add starting material at different points along the reactor's height also increases the selectivity of the process of testing increments of starting material with different fractional and chemical makeups. The new unit has a starting material capacity of 0.6 to 1 kg/h. The analysis block includes components that make it possible to subject liquid products and gases to chromatographic analysis and to perform a derivative thermogravimetric analysis of the coke content on a catalyst. Between 1.5 and 2 liters of catalyst may be used per loading. Gas dynamic indicators obtained for the new unit's operation were found to correlate with the analogous indicators of commercial catalytic cracking units. The new unit may

be used to research the following: the activity and selectivity of small batches of newly synthesized catalysts under conditions approximating the operation of large pilot units, the material balances and quality characteristics of various catalytic cracking products, the regeneration characteristics of various catalysts under different coke combustion conditions, the effect of residual coke on cracking indicators, the effect of different promoters on the secondary combustion of CO into CO₂ and the effect of activators and modifiers on the catalytic cracking process, and the effect of different factors on the catalytic cracking process both under conditions of an adiabatic decrease in temperature along the reactor's height and under isothermal conditions. Figure 1, tables 3; references 3: 2 Russian, 1 Western.

Base Oils From Hydrofined Raw Material

927M0015D Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 6, Jun 91 pp 8-12

[Article by A.N. Yershova, B.V. Gryaznov, M.M. Chernyshova, V.Z. Zlotnikov, S.A. Konakova, V.N. Bauman, and T.A. Dolbanova, Chemistry State Scientific Research Institute and Petroleum Refining All-Union Scientific Research Institute]

UDC 665.521.5:665.534

[Abstract] Because of their chemical makeup, the Western Siberian crude oils that are widely used in oil refining are less preferred as raw material for producing high-quality oils than are Volga-Ural crude oils. One possible way of improving the quality of the oils produced from Western Siberian crude oils is to develop and use processes to alter the chemical makeup and properties of the resultant procedures. Hydrogenation processes or, more specifically, hydrofining processes inscribed into the conventional oil manufacturing process flow are promising in this respect. The Omsknefteorgsintez Production Association has begun using a process to hydrofine viscous (at least 7.5 mm²/s at 100°C) oil distillate under soft conditions (temperature, 360 to 400°C; hydrogen pressure, ≈4 MPa) on a redesigned fuel hydrofining unit. The hydrofined distillate is processed in accordance with the conventional scheme, i.e., selective scrubbing with phenol, deparaffination, and hydrofining. The authors of the study reported herein compared the chemical composition and antioxidation and anticorrosion properties of component oils produced at the Omsknefteorgsintez Production Association by (1) the conventional process and (2) the newly developed process incorporating preliminary hydrofining. The prehydrofining oils had lower values for the following indicators: viscosity, density, molecular mass, specific variance of the color refraction index, and sulfur content. In addition, the prehydrofining oils had higher

viscosity index values than did the conventionally produced base oils, and their residual component was characterized by lower cokability, color, and sulfur content values. These changes were attributed to a change in the group chemical makeup of the oils during the hydrofining process. Specifically, hydrofining resulted in an increase in the content of naphthenic-paraffin hydrocarbons and a reduction in the content of aromatic compounds and tars. The increase in the viscosity index of the prehydrofining oils was attributed to an increase in the content of isoparaffin hydrocarbons after hydrofining. Hydrofining was also demonstrated to facilitate an increase in the content of monocyclic naphthenic-aromatic and aromatic hydrocarbons. Because hydrofining reduces the content of polycyclic aromatic hydrocarbons (which tend to undergo oxidation polymerization reactions), the prehydrofining oils were more resistant to oxidation than were the conventionally refined oils. When compared with conventionally refined oils, however, the prehydrofining oils had inferior anticorrosion properties with respect to lead. This fact was attributed to the higher content of naphthenic-paraffin hydrocarbons in the hydrofining oils and to the fact that the removal of organosulfuric compounds during hydrofining eliminates the anticorrosion protection afforded by them. With respect to copper, however, the anticorrosion properties of the prehydrofining oils were nearly identical to the conventionally refined oils. The research conducted thus confirmed that preliminary hydrofining can improve the quality of the Western Siberian crude oil generally used to produce base oils at the Omsknefteorgsintez Production Association. Tables 7; references 13: 12 Russian, 1 Western.

Alkyldiphenyl Oxides as a Base for Lubricating Oils

927M0015E Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 6, Jun 91 pp 21-22

[Article by V.V. Grigoryev, K.V. Prokofyev, and T.A. Bazylyuk, Kuybyshev affiliate, Petroleum Refining All-Union Scientific Research Institute]

UDC 665.765-404:621.892.28.001.57

[Abstract] Because of their superior (compared with naturally occurring oils) viscosity and low-temperature properties, resistance to oxidation, and improved lubricating and performance properties, synthetic oils are attracting ever-increasing attention in the Soviet Union and abroad. In view of this fact, the authors of the study reported herein examined the physicochemical and lubricating properties of selected alkyldiphenyl oxides and thickened specimens based on them. Alkyldiphenyl oxides with alkyl chains of different lengths were synthesized by alkylation of diphenyl oxide by C_7 through C_{10} alcohols on a heterogeneous zeolite catalyst in a tube-type flowthrough reactor at 150 to 160°C. The standard methods were used to determine their physicochemical

characteristics in comparison with analogous characteristics of dioctyl sebacate, which is used rather frequently as a lubricating oil component. The two were found to be quite close from the standpoint of their physicochemical properties. Studies of the antifriction properties of alkyldiphenyl oxides in a ball-plane friction pair on an SRV vibrotribometer at 20°C indicated that even under loads of 1,000 to 2,000 N, a lubricating film of the said compound remained rather high, and the friction coefficient remained rather low. The authors then proceeded to study the properties of alkyldiphenyl oxides thickened with polymer additives. These studies confirmed that alkyldiphenyl oxides mix well with polymethacrylates and polybutenes. It was discovered that between 10 and 20% polymer or polymer concentrate is required to obtain a viscosity of 9 to 11 mm^2/s at 100°C. The thickened specimens also proved to have a high viscosity index (125 to 155). Of the mixtures tested, alkyldiphenyl oxides plus polybutenes proved most interesting. To assess the potential of thickened alkyldiphenyl oxides as a base for industrial oils and hydraulic fluids, the authors used them as a base for preparing a prototype industrial oil with conventional antioxidation and antiviscosity additives. Data obtained from tests of the experimental oil indicated that it has good lubricating properties, is resistant to oxidation, and has a high viscosity index. The authors thus succeeded in confirming the possibility of using alkyldiphenyl oxides as a base for creating quality oils. Figures 3, tables 3; references 5 (Russian).

Hydrogenated Diaryl Alkanes as a Base for Lubricating Materials

927M0015F Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 6, Jun 91 pp 23-24

[Article by V.V. Grigoryev and G.V. Surovskaya, Kuybyshev affiliate, Petroleum Refining All-Union Scientific Research Institute]

UDC 665.658.2.094.17:665.656.6

[Abstract] Synthetic naphthenic hydrocarbons may be used to manufacture electrical insulation or as a base for hydraulic fluids or other lubricating materials. In view of the importance of naphthenic hydrocarbons, the authors of this study examined the hydrogenation of diaryl alkanes and the properties of the resultant hydrogenation products and their individual fractions. As starting materials, they used ditolylmethane and 1-phenyl-1-xylyl ethane. The hydrogenation was conducted in a continuous-type pilot unit with a 500-cm³ reactor made of 12Cr18Ni10Ti steel filled with packing and the catalyst AP-56. Throughout the process, the hydrogen:starting material ratio was kept at 1,000:1 (by volume), the pressure was varied from 3 to 5 MPa, the space velocity was varied from 0.5 to 1.5 h⁻¹, and the temperature was kept between 280 and 360°C. The composition of the resultant hydrocarbons was determined chromatographically by the simulated distillation method, and their

physicochemical properties were determined by the standard methods for lubricating oils. The experiments conducted established that (1) increasing the temperature to 340-350°C increased the content of hydrogenated hydrocarbons; (2) increasing the pressure facilitates the hydrogenation process; and increasing the space velocity at which the starting material is fed reduces the amount of products of complete hydrogenation of ditolylmethane. Further studies revealed that the viscosity of the hydrogenation products is higher and their density and drying temperature are lower than those of the starting diaryl alkanes. Hydrofining increased specific volumetric electrical resistance. The hydrogenated diaryl alkanes produced were tested at Riga Polytechnic Institute as high-traction lubricating materials for friction drives and variable-speed gears. The tests revealed that the friction coefficient of the new synthetic oils is 10 to 30% higher than that of a petroleum-based analogue. The new oils were found to have friction properties close to those of the oil Santotrac 50. The studies performed thus confirmed the promise of using products of the hydrogenation of diaryl alkanes as bases for lubricating oils. Figures 3, tables 2; references 6: 5 Russian, 1 Western.

Motor Oil Additives Based on Dithiophosphoric Acid and Unsaturated Compounds

927M0015G Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 6, Jun 91 pp 24-26

[Article by S.B. Borshchevskiy, I.S. Levitina, Ye.V. Shabanova, and G.G. Kotova, Petroleum Refining All-Union Scientific Research Institute]

UDC 665.7.038.5

[Abstract] The zinc dialkyldithiophosphates that are widely used as motor oil additives are plagued with a number of shortcomings, including high ash content. One way of developing ash-free additives may be to use esters of dialkyldithiophosphoric acids by reacting the respective acid with various unsaturated compounds. To determine the feasibility of this approach, the authors of the study reported herein synthesized esters of diisobutylidithiophosphoric acid. The starting acid was produced by the method of M.I. Kabachnik, and the structure of the acid synthesized was studied by infrared and proton magnetic resonance spectroscopy. The ester yields were found to be independent of the length of the hydrocarbon chain of the starting unsaturated hydrocarbon. The yields did rise, however, in the presence of an aryl substituent (styrene) and oxygen atoms (vinylacetate). In the former case, the ester yield proved to be higher when acrylic and methacrylic acids were used as the starting initial unsaturated compounds. All of the compounds synthesized proved to be soluble in mineral oil and to improve its tribological characteristics. Increasing the size of the alkyl residue in the ester molecule worsened the oil's tribological characteristics somewhat, however. Having an aryl substituent or oxygen atoms present in this residue increased the oil's

lubricating and anticorrosion properties. These findings led the authors to speculate that the higher the nucleophilicity of the double bond in the starting unsaturated compound, the higher the lubricating and antifriction properties of the resultant ester. The most effective esters produced were those obtained by reacting diisobutylidithiophosphoric acid with vinyl acetate or styrene. The most effective of the electrophilic compounds produces turned out to be the ester based on methacrylic acid. The research conducted was used as the basis for developing the ash-free additives BMA-5 (based on vinylacetate), TEF-3 (based on styrene), and ADF (based on methacrylic acid). From an effectiveness standpoint, all three additives are in no way inferior to the additive Mobilad DS-140 produced by the Mobil Oil Company. Figures 2, tables 3; references 5 (Russian).

Esters of Tetramethylolcyclopentanol as a Base for Lubricating Oils

927M0015H Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 6, Jun 91 pp 26-28

[Article by M.A. Mamedyarov and G.N. Gurbanov, Petrochemical Processing Institute imeni Yu.G. Mamedaliyev, Azerbaijan Academy of Sciences]

UDC 542.951.3:621.892.28

[Abstract] In a previous study, the authors of the present study examined and reported on the esters 2,2,6,6-tetramethylolcyclohexanol. In a continuation of that line of research, they now report on their synthesis and study of tetra- and pentaesters of tetramethylolcyclopentanol. They synthesized the said compounds by reacting cyclopentanol with paraform in an alkaline medium. During the synthesis, the alcohol:acid ratios were kept at 1:4.2 and 1:8, respectively, and n-toluene sulfo acid (1% of the total mass of the reagents used) served as a catalyst. The reaction was conducted at 170 to 210°C until the release of reaction water ceased (3.5 to 7 hours). The resultant esters were separated from the reaction mass by vacuum distillation. An ester yield amounting to 21.2-65.5% of the theoretical yield was achieved. The esters produced turned out to be colorless liquids with high boiling points. They were discovered to have a high viscosity at 100°C and have lower congelation and higher drying points than do the esters TMCH and pentaerythrite. They also proved to have satisfactory viscosity-temperature properties in the negative-temperature range. Studies of the tetramethylolcyclopentanol esters synthesized to determine their promise as bases for lubricating materials demonstrated that they have a very low evaporativity and a rather high thermooxidative stability. The tetramethylolcyclopentanol esters synthesized were tested as dispersing media for high-temperature plastic lubricants, compressor oils in the production of high-pressure polyethylene, and components for helicopter oils to improve their lubricating properties. These results led to authors to recommend

the new esters as a base for and component of lubricating oils. Tables 3; references 5: 4 Russian, 1 Western.

Polyhexene-1 as a Viscosity Additive to Lubricating Oils

927M00151 Moscow *KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL* in Russian No 6, Jun 91 pp 28-29

[Article by A.I. Akhmedov and E.U. Isakov, Additive Chemistry Institute, Azerbaijan Academy of Sciences]

UDC 678.741.524:621.892.8

[Abstract] Various polymers and copolymers of vinyl monomers have been investigated as potential viscosity additives for lubricating oils. The only ones that have gained popularity in the Soviet Union are polyisobutylenes and polyalkylmethacrylates. Polyisobutylenes are inexpensive but have proved to be inferior to polyalkylmethacrylates. The latter, while satisfactory from a performance standpoint, are plagued by the problems of high cost and limited availability. In an effort to meet the critical need for a viscosity additive that is based on an inexpensive and readily available material and that possesses good viscosity-temperature properties, the authors of the study reported herein examined polyhexene-1 as a viscosity additive for lubricating oils. They polymerized hexene-1 in the presence of aluminum chloride in a hexane solution in the temperature range from -10 to +20°C. After completion of the polymerization, the resultant polymer-in-hexane solution was treated with a 3% solution of alkali to deactivate the catalyst. The polymerization product was then rinsed with water and filtered, and the hexane was filtered off. The residue was then subjected to vacuum treatment to remove traces of solvent and the low-boiling fractions. The structure of the resultant polymers was subjected to infrared and proton magnetic resonance spectroscopic studies. An aluminum chloride concentration of 5% was found to be optimal. Temperature changes proved to have little effect on the yield of polymer; reducing the temperature from 20 to -10°C increased the molecular mass from 5,000 to 15,000. The nature of the monomer did prove to affect the course of the polymerization process. Studies designed to determine the feasibility of the study polymer as viscosity additives for lubricating oils revealed that polyhexene-1 is relatively more resistant to thermal breakdown than are currently used commercial viscosity additives. Polyhexene-1 proved to be superior to polyisobutylene with respect to improving the viscosity-temperature properties of the oil I-12A. A 3% content of polyhexene-1 in I-12A oil was found to

result in the best viscosity and viscosity index. Increasing the polyhexene-1 concentration to 5-7% increased viscosity but not the viscosity index, and the viscosity index began to drop with a 9% polyhexene-1 concentration in the oil. Figures 3, table 1; references 2 (Russian).

Determining Olefin Hydrocarbons in Petroleum Products by ^1H NMR Spectroscopy

927M00151 Moscow *KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL* in Russian No 6, Jun 91 pp 29-30

[Article by D.F. Kushnarev, V.M. Polonov, V.I. Donskikh, Ye.D. Kiryukhina, and G.A. Kalabin, Petrochemical and Coal Chemical Synthesis Institute, Irkutsk State University]

UDC 665.76.543.422.25

[Abstract] The performance characteristics of fuels are determined by their chemical makeup, specifically by their content of olefin hydrocarbons. The presence of a reactive double bond in olefin hydrocarbons significantly reduces the stability indicators of fuels, oils, and other commercial petroleum products. Chemical methods based on hydrogenation and ozonation reactions are primarily used to determine olefin hydrocarbons in petroleum products. The iodometric method is also popular. The most promising among instrumentation methods is that of nuclear magnetic resonance based on nuclei ^1H . This method is fast, universal, and generally free of the shortcomings plaguing chemical methods. In addition, only a small amount of specimen material (between 10 and 100 mg) is required for an analysis. In an effort to increase its use in the Soviet Union, the authors of the study reported herein set out to develop a universal method of determining the content of olefin fragments in petroleum products by using ^1H NMR spectroscopy. They present a series of equations to support the technique of ^1H NMR spectroscopy determination of olefin hydrocarbons. Specifically, they provide formulas for calculating the content of olefin hydrocarbons as a percentage of the total weighted portion, the mean molecular mass of the olefin fragment, and the number of moles of olefin fragments. They also provide a series of expressions that may be used to compare the results obtained by using ^1H NMR spectroscopy with those obtained by using the iodometric technique. The relative errors of ^1H NMR spectroscopy and iodometric determinations of the olefin hydrocarbons in 23 petroleum product specimen were compared and are presented in table form. The maximum error of the determination method presented was found to amount to 10% (rel.). Table 1; references 5: 4 Russian, 1 Western.

Heat-Resistant Unsaturated Polyamides

927M0023G Novosibirsk *IZVESTIYA SIBIRSKOGO OTDELENIYA AKADEMII NAUK SSSR: SIBIRSKIY KHMICHESKIY ZHURNAL* in Russian No 3, May-Jun 91 (manuscript received 5 Jul 90) pp 112-115

[Article by V.V. Nikiteyev, Yu.Ye. Doroshenko, D.M. Mognonov, A.A. Iznyeyev, and Ya.G. Urman, Buryat Natural Sciences Institute, Siberian Department, USSR Academy of Sciences, and Buryat Agricultural Institute, Ulan-Ude]

UDC 678.675

[Abstract] In view of the critical need to develop polymers combining such qualities as high heat resistance and ease of processing into products by currently existing commercial methods, the authors of the study reported herein worked to synthesize heat-resistant, soluble unsaturated polyamides capable of forming cross-linked three-dimensional products with a high heat resistance and good mechanical and electrical indicators. To synthesize the said unsaturated polyamides, the authors used a low-temperature reaction of polycondensation of dichloroanhydride of itaconic acid with aromatic diamines in a solution of aprotic amido-type solvents. As the aromatic diamines, the authors used 4,4'-diaminodiphenylmethane, 4,4'-diaminodiphenyl oxide, m-phenylenediamine, and n-phenylenediamine. The resultant polymers were finely dispersed yellow-brown powders that were soluble in amide solvents; concentrated formic, chloroacetic, and fluoroacetic acids; dimethylsulfoxide; and sulfolane. The structures of the resultant unsaturated polyamides were confirmed by elemental analysis and infrared and ^{13}C nuclear magnetic resonance spectroscopy. Differential thermal analysis of the study unsaturated polyamides revealed an exothermal peak in the temperature range from 473 to 493 K that was explained by polymerization along the C-C bonds. Thermal-oxidative destruction of the solidified polymer (accompanied by the release of volatile products with a high exothermal effect) was found to occur at 623-673 K. The study unsaturated polyamides were used to produce unfilled plastics with a good set of physicomechanical properties. The plastics were produced by direct pressing at 493 to 523 K and a specific pressure of 10 to 20 MPa. The new plastics were found to be much harder than commercial aliphatic and aromatic polyamides and, consequently, have a low impact viscosity. Their breaking stress during bending of the plastics based on 4,4'-diaminodiphenyl oxide and dichloroanhydride of itaconic acid is comparable with the indicators of aliphatic polyamides. During aging tests conducted at 573 K in air, the unsaturated polyamides' breaking stress during bending did not decrease even after 100 hours. This fact favorably distinguished the new unsaturated polyamides from commercially produced polyamides, which experience a 75 to 80% loss of strength at temperatures as low as 423 K. Despite the fact that the new unsaturated polyamides are standard polar polymers, they have a dielectric constant of about

2 at 293 K (which is characteristic of most nonpolar polymers). The new unsaturated polyamides thus appear promising for use in producing heat-resistant construction materials capable of withstanding moderate mechanical loads within a broad temperature range and possessing dielectric properties that permit their use as heat-resistant, high-quality, low-frequency dielectrics. Figures 2, tables 4; references 10: 7 Russian, 3 Western.

Controlling the Ionic Permeability of Adherent Polymer Materials

927M0024I Leningrad *ZHURNAL PRIKLADNOY KHMII* in Russian Vol 64 No 3, Mar 91 (manuscript received 10 May 90) pp 636-640

[Article by N.D. Sakhnenko, A.P. Pospelov, M.V. Ved, and T.P. Yaroshok, Kharkov Polytechnic Institute imeni V.I. Lenin]

UDC 620.198

[Abstract] Ionic permeability is one of the most important parameters characterizing the protective ability of polymer coatings. Because quantitative information regarding ionic transport in the system metal-coating is extremely important to reliable prediction of a coating's useful life under actual operating conditions, the authors of the study reported herein conducted a study to establish a functional connection between the parameters of the ionic permeability of adherent polymer materials and the electrochemical characteristics of the system metal-coating. The studies were conducted on a system of protective coatings on divinyl-acetylene (EKZhS-40 and KhV-5153), vinyl (VL-02, KhS-413, and KhV-5153), epoxy divinyl-acetylene (VL-02, EP-755, and KhV-5153), and epoxy (B-EP-0126) bases. Free films and coatings were applied to 08 kp steel in accordance with the standard technology. The permeability of the polymer materials for chloride ions was determined by analysis of the system's electrochemical response to a gradual change in the concentration of the sodium chloride solution. The temperature was maintained at 25 \pm 0.5°. A VK-2-16 high-ohm voltmeter was used to measure the potentials of the electrodes, and an R-5083 automatic alternating-current bridge was used to measure the impedance. Potentiometric titration was used to study the transfer of chloride ions in the free films. Analysis of the potentiometric measurement results established that increasing the concentration of the sodium chloride solution results in a shift of the potential of electrodes with a coating to the side of more negative values. The potential of electrodes with a coating is the result of at least two competing processes, i.e., activation of the metal surface due to an increase in the number of chloride ions and formation of passivating layers. Six expressions are provided for determining the ionic permeability of adherent polymer coatings. A comparison of the values of the parameters of chloride ion transfer in free films obtained by the impedance and potentiometric titration methods demonstrated that the

approach of using electrochemical measurements as a basis for estimating the ionic permeability of adherent coatings does produce reliable results. This finding led the authors to recommend their new approach for use in continuous nondestructive testing of the corrosion resistance of construction materials. Figures 4, tables 2; references 7 (Russian).

High Strength High Modulus Carbon Fibers

927M0033A Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 5-8

[Article by M. T. Azarova]

UDC677.529:677.494.745.32

[Abstract] High strength carbon fibers having high modulus of elasticity are made chiefly from polyacrylonitrile (PAN). In the USSR PAN-based carbon fibers are used in such high strength materials as LU-P, ELUR P, ELUR-0085, and UKN-P, and in high modulus materials as Grapit-P, Kulon-P filament, Kulon and Kulon-P tape. All high strength materials are produced industrially, while the high modulus materials are in experimental production. High strength fillers are produced in two textile forms, either uni-directional tape (250-260 mm wide) or 90 mm non-twisted braid (UKN-P, Granit-P, and Kulon-P). The tape is used primarily for lay-up work, while the braid is used chiefly for wrappings to produce pre-impregnated fabrics. Tapes LU and ELUR have strengths of 2.5-3.2 GPa and elasticity modulus of 220-270 GPa. Other properties such as linear density of filaments, specific resistance, tensile strength, compression strength, and modulus of elasticity are listed in a table for each of the above materials. Prerequisites for shaping the structures and properties of PAN-based carbon fibers are discussed. Figures 3; references 12: 5 Russian, 7 Western.

Basic Trends in Research on Preparation of Carbon Fiber Materials from Cellulose Hydrate Fibers

927M0033B Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 8-10

[Article by M. Ye. Kazakov, VNIIPV]

UDC677.529.021.12:677.463

[Abstract] Although production of carbon fiber materials from cellulose hydrate fiber in foreign countries has been cut back greatly (for ecological reasons), research on the production and properties of this material continues. Production of cellulose hydrate fiber as a source for carbon fiber production in the USSR may be justified from the standpoint that it relies on a renewable resource (wood), and that the carbon fiber materials made from it have unique properties. The cutback in demand for carbon fiber materials associated with the recent conversion of defense-oriented industries is evidently the result

of various branches of the national economy not being ready to assimilate these materials. However, after re-orientation in 1995-2000, it is estimated that demand for carbon fiber materials will rise again. The VNIIPV has developed and introduced a wide assortment of carbon fiber materials based on viscose fiber. Basic trends in research on preparation of carbon fiber materials from cellulose hydrate fiber lie in improving the quality of viscose fiber, resolving ecological problems associated with its production, optimization of production technology to improve properties and lower costs, develop new high grade carbon fibers, and search for new applications. References 16: 11 Russian, 5 Western.

Carbon Fibers from Petroleum Isotropic Pitch

927M0033C Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 10-13

[Article by V. Ya. Varshavskiy and A. Ya. Lyalyushkin, VNIIPV]

UDC677.529.021.12:662.749

[Abstract] Some advantages of producing carbon fibers from petroleum pitch instead of polyacrylonitrile or viscose fibers, consist of fewer process steps and the efficiency derived from forming the fiber directly from a melt and with less harmful wastes. In the present work basic features of producing an isotropic petroleum pitch from which carbon fiber can be produced are discussed. Data are presented which confirm the feasibility of using such fibers in various applications in the national economy. Figures 2; references 14: 6 Russian, 8 Western.

Carbon-Carbon Composite Materials

927M0033D Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 13-15

[Article by N. P. Radimov, VNIIPV]

UDC661.666:677.529

[Abstract] Carbon-carbon composite materials consist of carbon fibers originating from various textile structures and carbon matrices derived from coke, polymeric resins, pitch, and pyrolytic carbon. These composites have high heat and chemical resistance, electrical conductivity, friction and anti-friction properties as compared to special steels, alloys, graphite, or polymeric composites. New items fabricated by a new technology have been tested for heat resistance and chemical erosion during abrasion at 2000-2500 K and 10-12 MPa. Objects fabricated from the above composites having a variety of geometric shapes are highly conductive electrically and resist abrasive action. Despite their high cost, carbon-carbon composites are finding applications as components in new technology for which there are no alternatives in regard to chemical and ecological purity, e.g. chemical machine building, automobiles, tractors, and nuclear energy. These composites are expected to be

widely used as endoprosthetics, and in stomatology. References 5: 2 Russian, 3 Western.

Synthesis of High Molecular Weight Acrylonitrile Copolymer and Making Fiber from It

927M0033E Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 18-19

[Article by I. A. Barsukov, G. N. Khusnulin, A. T. Serkov, V. A. Dmitriyev, and N. P. Kruchinin (deceased), Chemistry SRI at Nizhegorodskiy State University; VNIIPV]

UDC678.745.32-13.02+677.494.745.32.021.125.2

[Abstract] The molecular weight of a polymer has a significant effect on the chemical, physical, and mechanical properties of any fibers made from it. The molecular weight of polyacrylonitrile used in the USSR for making fibers designated for construction purposes, ranges from 55,000 to 65,000. Fibers made from this polymer do not have very high mechanical properties. Further increase in strength is possible only by using higher molecular weight polymers, 100,000 to 500,000, as confirmed by the patent literature. In Japan, high molecular weight polyacrylonitrile is produced by suspension polymerization, while at the Nizhegorodskiy State University a method was developed for continuous production of rayon spinning solution of high molecular weight polyacrylonitrile. Other data attest to the possibility of increasing the strength characteristics of polyacrylonitrile fiber produced by the salt method. Figures 3; references 3 (Russian).

Analysis of Structure and Viscous-Elastic Properties of Technical Grade Polyacrylonitrile Braids

927M0033F Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 22-23

[Article by B. M. Tarakanov, Ye. S. Gromova, A. M. Stalevich, M. T. Azarova, N. A. Shepeleva, and L. M. Ganchuk, LITLP]

UDC677.494.745.32:539.26

[Abstract] Uncovering the relationship between the mechanical properties and the structural parameters of polyacrylonitrile braids, whose properties largely determine the quality carbon fibers produced from them, is a vital technological task and may serve as a basis for developing quality control methods. In the present work a study was made to establish a correlation between the deformation properties of polyacrylonitrile braid and the parameters of their sub-molecular structures. This not only has applied significance, but is also intrinsically interesting from the standpoint of building a structural model simulating the entire complex of viscous-elastic properties of oriented polymer systems. X-ray analysis established that polyacrylonitrile braids from various

production lots have different sub-molecular structures. In samples having a low degree of crystallite disorientation in relation to the texture axis, the non-ordered region has a greater density, and crystallites have a lesser density, than samples having a higher degree of crystallite disorientation. There is a correlation between the sub-molecular structure of polyacrylonitrile braids and the process parameters used in preparing them. With this correlation, it becomes possible to make a structural interpretation of the viscous-elastic properties and it may also be used in the analysis of polyacrylonitrile braid production. References 7: 6 Russian, 1 Western.

Some Causes of Unstable Thermal and Thermo-Mechanical Properties of Polyacrylonitrile Fibers

927M0033G Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 2526

[Article by V. V. Kochetkov, N. A. Shepeleva, T. K. Mikhaylova, L. M. Ganchuk, M. T. Azarova, N. V. Kochetkova, and V. A. Medvedev, VNIIPV]

UDC677.494.745.32.021

[Abstract] Since the mechanical characteristics of carbon fibers are largely governed by the properties of the initial polyacrylonitrile fibers from which they are made, it may be assumed that the unstable mechanical properties of carbon fiber UKN/5000 are associated with the instability of a number of characteristics in the initial polyacrylonitrile fiber. This may be further exacerbated during the heat treatment process. The temperature-deformation conditions under which the fibers are treated is known to be a major technological parameter, and by knowing the deformational properties and thermal conversions of polyacrylonitrile fiber at this stage, it is may be possible to optimize the oxidizing parameters and obtain carbon fibers having maximum strength (for a given defect level in the initial polyacrylonitrile fiber). In the present work a study was made of the stability of thermal and thermo-mechanical properties of industrial grade polyacrylonitrile fiber having a linear density of 850 tact produced by the thiocyanate method. This fiber has considerable instability in thermal and thermo-mechanical properties. Instability in such parameters as itaconic acid content in the polymer, temperature and concentration of the settling bath, and pull time evidently has a major effect on stability of the thermal and thermo-mechanical properties. Stabilization of these parameters could conceivably decrease the instability in the mechanical properties of the carbon fibers. Figures 3; references 3: 1 Russian, 2 Western.

Structure of Carbon Fibers

927M0033H Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 26-29

[Article by G. B. Skripchenko, Mined Fuels Institute]

UDC677.529.014.8

[Abstract] Carbon fiber is a unique material combining the special features of a graphite layer, two-dimensional order in the laminates, and fiber structure. In the present work the properties of carbon fibers are considered from the point of view of a carbon stripe (analogous to carbon chain) orientated amorphous-crystalline polymer being in the glassy state at temperatures below 2300 K. Characteristic regions in the formation of carbon fiber structures are identified and the correlations between structure parameters established. The nature of the change in the axial component of texture with change in treatment temperature, and its relation to elasticity and strength of the fiber, are described. Figures 5; references 13: 5 Russian, 8 Western.

High Modulus High Strength Carbon Fiber from Polyacrylonitrile

927M0033I Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 29-30

[Article by V. A. Mikhaylova, N. A. Savostyanova, N. V. Bondarenko, A. V. Khorkhorin, and V. M. Chermashentseva, Electro-Carbon Components SRI (A-U), Elektrotrougli]

UDC678.746.32

[Abstract] High modulus carbon fibers (modulus of elasticity 600-800 GPa) may be produced by boronizing the fiber during heat treatment at 2700-2900° C, although in most cases the resulting fiber has low pull strength (below 2 GPa). This is evidently related to structural changes in the boronized fiber. In the present work high strength and high modulus carbon fiber was prepared from polyacrylonitrile subjected to various pre-treatment stages prior to boronizing at 2800° C, viz. thermo-oxidation, oxidation and carbonization, oxidation, and carbonization and graphitization at 2100° C. It was demonstrated that both the preliminary method of heat treatment and the structure of the fiber being boronized affect the strength of high modulus carbon fiber. A more oriented structure in the fiber being boronized, and relaxation of stresses during the heat treatment process enhance the strength of high modulus carbon fiber to 3.0-3.5 GPa at over 700 GPa elasticity modulus. Figures 3; references 2: 1 Russian, 1 Western.

Pyrolysis of Polyacrylonitrile Fibers Subjected to Laser Radiation

927M0033J Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 30-31

[Article by N. V. Platonova, I. B. Klimenko, B. M. Tarakanov, B. A. Vinogradov, K. Ye. Boyarkin, S. P. Mayburov, and M. L. Surkina, Textile and Light Industry Institute imeni S. M. Kirov, Leningrad]

UDC677.494.745.32.021.125.5:539.1.043+677.529.021.12

[Abstract] An important but lengthy and energy consuming stage in the production of polyacrylonitrile based carbon fibers is oxidative stabilization. In the present work the feasibility of shortening this stage by preliminary CO₂-laser radiation is examined. The results show that the radiation of technical grade braided fiber initiates cyclization processes during subsequent heat treatment and cuts the oxidation time. It also lessens the cyclization initiation temperature, the heat effect corresponding to the process, and loss in weight. References 4 (Russian).

Properties of Heat Stabilized Polyacrylonitrile Filaments and Their Applications

927M0033K Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 32-33

[Article by A. K. Iyeleva, M. T. Azarova, Ye. G. Budylina, and V. V. Kochetkov, VNIIPV]

UDC677.494.745.32-96.014/017+677.494.745.32-96.004.14

[Abstract] The demand for materials having high thermo-oxidative resistance continues to grow with the development of technology. At the present time oxidized filaments based on polyacrylonitrile called, "Panox", are being widely used abroad. These filaments neither burn nor decompose in air at temperatures to 250-260°C. An 8-10 percent elongation to breaking value facilitates their use in conventional weaving equipment. Fabric made from Panox filaments is used for the filtration of hot gases, petroleum products, acids, and in combination with natural fibers for making protective clothing for welders, metallurgists, and firemen. They exceed the properties of asbestos in many respects as well as being much lighter in weight. An analogous product, "NTS", was also developed at the Scientific Production Association "Khimvolokno" and its specifications are presented. Figures 3; references 4: 2 Russian, 2 Western.

Super-Molecular Structural Features of Isotropic Petroleum Pitch and Carbon Fiber Derived Thereof

927M0033L Mytishchi KHIMICHESKIYE VOLOKNA
in Russian No 3, May-Jun 91 pp 38-41

[Article by V. Ya. Varshavskiy, M. L. Uchitel, L. P. Braverman, V. A. Platonov, and T. Ye. Khaplanova, VNIIPV]

UDC662.749:539.2+677.529.014.8

[Abstract] Fiber-forming petroleum pitches are normally obtained by thermopolycondensation of native or pyrolytic resins. During the course of this process, at the stages preceding formation of meso-phases, polycyclic

aromatic compounds are formed whose molecules contain small quantities of aliphatic groups. Polycyclic molecular structure is even more characteristic for the fraction that is insoluble in hydrocarbons, since it is the product of deeper polycondensation. Neither the sub-molecular formations in pitches not containing a meta-phase, nor the carbon fibers based on them have yet been studied. In the present work rheological analysis and electron microscopy were used to demonstrate the presence of anisotropic super-molecular formations in isotropic pitch and carbon fibers made from it. The genesis and development of these formations are influenced by the electron structure of the pitch molecules, its fractional composition, and the terminal temperature of fiber formation. Figures 4; references 7: 4 Russian, 3 Western.

Carbon Fiber Material Having Anti-Oxidizing Protective Coatings

927M0033M *Mytishchi KHIMICHESKIYE VOLOKNA*
in Russian No 3, May-Jun 91 pp 47-48

[Article by L. M. Mizayeva, N. P. Radimov, T. K. Mikhaylova, and P. F. Lyasota, VNIIP]

UDC677.529.021.125:677.865.11

[Abstract] Heat and chemical resistant materials that maintain their service characteristics in corrosive media over long periods of time at temperatures of 350-600°C are needed for further developments in the space, automotive, and chemical machine building sectors of the national economy. These materials are composites of metallic, ceramic, and carbonaceous materials reinforced with carbon fibers. Although carbon fibers have many unique properties, they also have some undesirable features such as rapid oxidation in air starting at 350°C and causing a depreciation in physical properties. Also, carbon fiber surfaces react with various matrices, especially metals at the fiber-metal interphase. Barrier coatings consisting of titanium or silicon carbides may be applied to carbon fiber

materials to increase thermo-oxidative resistance and improve elastic-strength and adhesion properties. Reagents used are silicon and titanium chlorides, colorless liquids with low boiling points making them suitable for gas phase deposition as coatings at 1300-1350°C. In the present work such as gas phase method was developed for applying single and multi-component carbide coatings on various carbon fiber textile materials. While maintaining strength and elastic qualities, the thermo-oxidative resistance of the carbon fiber material is also increased, and the temperature range at which decomposition takes place is shifted by 150-250°C to a higher range. Coated carbon fiber materials are increasingly used to fabricate filters for hot dust, gas, and air mixtures. Figure 1; references 5 (Russian).

Development of Light-Weight Respirators Using Carbon Fabrics

927M0033N *Mytishchi KHIMICHESKIYE VOLOKNA*
in Russian No 3, May-Jun 91 pp 51-52

[Article by L. Yu. Kudryavtseva, M. L. Shkrabo, V. I. Savin, Ye. A. Farberova, M. Ye. Kazakov, Yu. F. Gridina, and L. K. Borisova, VNITIUS]

UDC677.529-96.004.14:614.894

[Abstract] Filter type respirators SH-1, "Lepestok", and "Kama-200" are successfully used in various industries. However, the work areas in many enterprises contain such dangerous vapors as benzene, acetone, chlorine, sulfur dioxide, hydrogen sulfide, and hydrogen chloride, as well as ammonia in concentration 5-10 times above the allowable limit. The respiratory organs of employees may be protected from low concentrations of pollutants by producing light-weight gas-dust respirators that are based on durable and elastic sorbent materials, such as activated charcoal fabrics. In the present work Ural and UVK activated charcoal fabrics were used in the design of respirators for protection against ammonia and sulfur dioxide which have active lifetimes of 2 and 5-6 hours, respectively. References 5 (Russian).

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